

Final Report

Montrose Chemical Superfund Site, Los Angeles County, California

One-Dimensional Thermal Remediation Treatability Study

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Introduction

Thermal remediation is being evaluated for dense non-aqueous phase liquids (DNAPL) in the subsurface for the Montrose Chemical Superfund Site, particularly at the Montrose Chemical Corp of California (Montrose) former plant property (“plant property.”) A feasibility study is underway to evaluate alternative approaches for DNAPL recovery and/or destruction, in accordance with EPA’s Record of Decision for the Dual Site Operable Unit, issued in 1999 (ROD). The ROD selected a remedial action for groundwater at the Montrose and Del Amo Superfund Sites, and required a second phase of remedy selection to address DNAPL. DNAPL recovery or destruction could provide benefits including, but not limited to reducing the risk of DNAPL migration, as well as the long-term uncertainty of the groundwater remedy.

The DNAPL at the former Montrose plant property is comprised primarily of chlorobenzene with large concentrations of co-solvated DDT (dichlorodiphenyltrichloroethane) and its degradation products DDD (dichlorodiphenyldichloroethane) and DDE (dichlorodiphenyldichloroethylene). The purpose of this treatability study was to evaluate the effectiveness of thermal remediation technologies such as steam injection or electrical resistance heating (ERH) to recover chlorobenzene from the site soils. Specifically, the bench scale tests that were performed estimate the amount of steam or energy that are required to recover chlorobenzene, and indicate residual contamination levels that might be present after thermal remediation. An additional purpose was to determine the fate of DDT in the DNAPL in a thermal remediation. Unless otherwise noted, the term DDT or total DDT will be used to refer to the sum of DDT, DDE and DDD concentrations.

The treatability study consisted of measurement of the physical properties (density, viscosity, surface and interfacial tensions) of site DNAPL as a function of temperature in the range of 10°C to 90°C, and performing one-dimensional steam injection experiments on contaminated site soils. Knowledge of the physical properties of the DNAPL as a function of temperature allows a greater understanding of the fate of DNAPL during thermal remediation. The one-dimensional steam injection experiments allow estimation of the recovery rate of chlorobenzene as a function of the number of pore volumes of steam (or energy) injected, the residual soil concentrations of the contaminants after thermal remediation, and estimates of the aqueous concentrations of the contaminants before and after steam injection. These one dimensional steam injection experiments also allow for gathering initial information on the fate of DDT during thermal remediation.

This data will determine whether thermal remediation would be effective for recovering chlorobenzene DNAPL and reducing the mass of chlorobenzene entering the downgradient plume at the Montrose Chemical Site and thus should be evaluated as a viable alternative during the Feasibility Study. This data does not, however, fully address the question of the nature and degree of DDT DNAPL mobility after the chlorobenzene has been volatilized from the DNAPL. The behavior of DDT DNAPL is being addressed by laboratory work done by Dr. Kent Udell on behalf of Montrose Chemical Corporation (Montrose).

Background. The Montrose Chemical Superfund Site is located in Los Angeles County, California (near Torrance). This former DDT manufacturing facility has subsurface DNAPL contamination consisting of primarily chlorobenzene and DDT.

The stratigraphy beneath the former Montrose plant property, starting at land surface, consists of fill material, the recent Playa deposits, the Palos Verdes sand, the upper Bellflower

aquitard, the Gage aquifer, an unnamed aquitard, and the Lynwood aquifer. A significant fraction of the DNAPL mass is believed to be in the Palos Verdes sand and upper Bellflower aquitard; thus the treatability study focused on the lower-permeability upper Bellflower aquitard materials. The first groundwater beneath the site is encountered within the upper Bellflower aquitard at a depth of approximately 60 feet below land surface. The upper 25 to 28 feet of the Bellflower aquitard is predominantly fine-grained sand with occasional discontinuous silty sand and silt interbeds. The lower 22 to 25 feet consists of a heterogeneous mixture of fine-grained sand, silty sand, fossiliferous sand, and silt. These sediments are interbedded, discontinuous, and vary in thickness, ranging from 0.1 to 4 feet thick. More details on the stratigraphy of these units beneath the former Montrose plant property can be found in Hargis + Associates (2004).

Thermal Remediation. Steam injection has been used as an enhanced oil recovery method since the 1930s, and its use for soil and aquifer remediation was pioneered in the 1980s by Dr. Kent Udell. Initial steam injection experiments in the laboratory showed that steam could effectively recover volatile contaminants such as gasoline constituents and organic solvents from one dimensional soil columns (Hunt et. al, 1988). Field trials and full-scale remediation have demonstrated the enhanced recovery of chlorinated solvents (Udell and Stewart, 1989), gasoline (Newmark, 1994), JP-5 (Udell et. al, 1994), and creosote (remediation performed by Southern California Edison at Visalia, CA) when steam injection is used in conjunction with vapor and groundwater extraction.

Initially when steam is injected into the subsurface, the steam condenses, and the latent heat of vaporization from the water heats the formation. When the formation reaches steam temperature, a steam zone forms in the subsurface. Water and liquid contaminants contained in the subsurface are pushed ahead of the steam zone as it grows, displacing them towards recovery wells. The displacement process is aided by the reduction in viscosity of liquids as the temperature increases. Volatile contaminants are vaporized and recovered in the vapor phase, or they may recondense at the front of the steam zone and be displaced ahead of the steam front and recovered as a liquid. The increase in temperature to steam temperature also increases the solubility of the contaminants in water, allowing recovery of a portion of the contaminants in the aqueous phase. Theoretical studies as well as laboratory and field experience have shown that when the soil is at steam temperature, reducing the pressure allows greater vaporization of volatile organic contaminants and thus greater vapor recoveries. Reductions in pressure can be brought about by halting steam injection while continuing to extract liquids and vapors. This is referred to as steam cycling, and is an important operational technique when steam injection is used for remediation purposes (Davis, 1998).

Electrical resistance heating (ERH) is another thermal remediation technology that has been found to be effective for the recovery of volatile contaminants from soils and groundwater. This technology was developed by Battelle Memorial Institute for the U.S. Department of Energy. Electrodes are installed in the subsurface, and electrical energy is applied between them. Although soils do not carry electrical current, the water in the pore spaces will carry the current between electrodes. The natural resistivity of the soils causes the energy to be dissipated as heat, and soils and groundwater are brought to the boiling point of water. Volatile contaminants are recovered in the vapor phase using soil vapor extraction, and liquids (contaminated groundwater and NAPLs) can also be extracted (Beyke et. al, 2000).

Laboratory treatability studies for the recovery of contaminants from soils using steam injection were developed by Dr. Udell. Experience with the results of these treatability studies

and the results of steam injection remediation in the field have shown that these one-dimensional steam injection experiments can predict fairly well the residual saturation of the contaminant that is likely to be left in the soil after a steam injection, and can indicate how much steam may be needed to reach that point. Parallels between the steam injection and electrical heating remediation technologies when liquid recovery is used with electrical heating allow the results of the steam injection tests to also predict what might be the outcome from an electrical heating remediation when the system is operated to also recover liquids.

Experimental Methods and Materials

Soil samples for the treatability study were collected during the DNAPL Reconnaissance Investigation performed by Montrose under EPA oversight in November 2003. A Rotasonic drill rig was used to collect a 4 inch diameter core. Cores 2 to 10 feet in length were obtained and extruded into plastic bags. Soil samples with visible DNAPL present were collected from boreholes SSB-2, SSB-6, PSB-4, and PSB-15. Most of the soil samples with visible DNAPL were collected from depths between 75 and 91 feet bgs. The soil samples were collected in 4 ounce glass jars. The jars were filled completely to reduce headspace, placed in a plastic bag, and then stored on ice until being shipped to Kerr Lab. At Kerr Lab, the soil samples were stored at 4°C until being packed into the column for the experiment.

DNAPL samples were collected from existing wells UBT-1, UBT-2, UBT-3, and UBE-1 on November 6, 2003. The DNAPL was collected in 1 liter amber glass jars. Groundwater collected from the same borehole as the DNAPL sample was added to the top of the sample jars to minimize vaporization of the DNAPL constituents. The jars were then shipped to Kerr Lab via overnight mail, where they were stored at 4°C.

Density Measurements. Density as a function of temperature for the DNAPL was measured by a method which follows ASTM D1217-93, as outlined in the RS Kerr Laboratory standard operating procedure (SOP), RSKSOP-289 (Appendix B). Measurements are made using 25 ml (± 0.03 ml) volumetric flasks suspended in a water bath. Density measurements are always made in triplicate. Measurements are started by determining the empty weight of the flask to the nearest 0.01 gram. The flasks are then filled to below the volumetric line with the fluid, and suspended in the water bath. The liquid is allowed to equilibrate initially to the highest temperature at which density measurements are to be made (in this case, 90°C). After the liquid has equilibrated to temperature, the volume is adjusted to the volumetric line with liquid that is also at the measurement temperature, and the flasks are then resuspended in the water bath to ensure that their contents are still at the desired temperature. After this second equilibration period, the volume in the flasks is double checked, and if needed volume adjustments are made. Once the volume is correct, the flasks are thoroughly dried on the outside, and on the inside of the flask above the volumetric line, then weighed to the nearest 0.01 gram. The weight of fluid in the flask and the volume (25 ml) are then used to calculate the density of the fluid in grams per milliliter. The temperature in the water bath is then adjusted for the next measurement, and the flasks resuspended in the bath. By working from the highest to the lowest temperature for which measurements are to be made, with each volume adjustment fluid is added to the flasks, and volumes are easily adjusted. Experience with this method has shown that the standard deviation of the three replicate measurements is generally about 0.001 gm/cm³.

Viscosity Measurement. The viscosity of NAPL samples was measured using a Brookfield Rotational Viscometer Model DV-I by a method that was equivalent to ASTM D2983 (see RSKSOP-273 in Appendix B), which also uses a Brookfield rotational viscometer. A different range of temperatures were measured in this experiment as compared with ASTM D2983, and a cabinet to maintain the entire apparatus at the desired temperature was not used. Instead, the small sample adapter with its water jacket assembly was used to hold the sample at the desired temperature for the viscosity measurement. The water jacket was hooked up to a water bath so that the temperature could be adjusted in the range of 10 to 90°C. Viscosity measurements were made in triplicate at 10°C increments at all viscometer rotational speeds that were appropriate. The accuracy of the measurements is generally 1 percent of the scale. Standard deviations for the repeat measurements have been found to be less than 0.05 dynes/cm.

Surface and Interfacial Tension Measurements. The surface and interfacial tension of NAPL samples and groundwater from the site were measured using a Fisher Surface Tensiometer, Model 20. The procedure follows ASTM Method D971. The one significant difference is the use of a water jacket and water bath to adjust the temperature of the liquids to the desired temperature (see REKSOP-274 in Appendix B). The samples were set into a jacketed beaker connected to a water bath to bring the fluid(s) to the desired temperature. Surface and interfacial tension measurements were made at 10, 50 and 90°C. Standard deviations of surface tension measurements have been found to be less than 0.4 dynes/cm.

Steam Injection Experiments. The apparatus for the one dimensional steam injection experiments is shown in Figure 1. Water was pumped by metering pumps connected in parallel to a steam generator. The steam generator was set to produce steam at approximately 150°C, and the temperature measured by a thermocouple at the effluent of the generator. The steam entered the top of a galvanized steel column that is two inches in diameter and six inches in length. The bottom column endcap contained approximately a one inch depth of clean sand, and the column was then packed with contaminated soils from the site, and capped at the top end. Prior to steam injection, an initial leachate sample was generated by filling the column with water and allowing approximately 24 hours for the water and soil to equilibrate before draining the water and submitting it for analysis as the initial leachate sample.

The temperature in the column during the steam injection was measured by thermocouples inserted into the top endpiece and at the center of the column. The column was wrapped with heater tape and then insulation to maintain steam temperatures throughout its length. The effluent line at the bottom of the column was a copper tube with a pressure gage and thermocouple, a valve, and then a heat exchanger to condense the effluent before it entered a sample container immersed in an ice bath. The thermocouples were attached to a computer which records the temperature of the system every 10 seconds throughout the experiment.

Steam was injected at a rate of approximately 75 ml/hour, which produced a flux of 23 cm/hr (0.40 cm/min). After steam breakthrough at the bottom of the column had occurred, steam cycling was performed by closing the effluent valve to build up steam pressure in the column, then opening the valve to release the pressure. In this manner, the pressure and temperature in the column can be increased to values closer to what might be used in the field. Several steam cycles were applied during the later part of the experiment. The final phase of the experiment was the collection of a final leachate sample, in which the column was filled with water, and contaminant concentrations in the steamed soil and water were allowed to equilibrate for approximately 24 hours. The contact time of the water with the soil was recorded in the lab

book. Then the leachate was drained from the column and submitted separately for analysis. Finally, the soil was removed from the column in sections for the collection of steamed soil samples for the analysis of remaining contaminants.

All of the effluent from the columns was collected throughout the steam injection experiments for analysis by a modification to EPA Method 8270 for chlorobenzene and DDT. Effluent produced by the injection of each pore volume of steam was collected separately. A total of approximately four pore volumes of steam were injected. The columns used for these experiments yield a pore volume of approximately 100 cm³. The duration of the experiments was approximately 5.3 hours.

Experience with previous steam injection treatability studies has shown that the flow rate used does not significantly impact the results when the column is oriented vertically (i.e., vertical downward flow is used). However, it is essential to ensure that a steam front is established in the column. Establishing temperatures greater than 100°C in the column does not necessarily establish a steam front, as pressures within the column may be great enough to condense the steam. When this occurs, the result is a hot water flood. Previous experience has shown that a hot water flood may not be effective for recovering contaminants from soils, while a steam flood using the same soils and contaminants may recover a large percentage of the contaminants. Thus, it is essential that a steam flood is produced. The production of a steam flood was confirmed by monitoring the mass of effluent collected and comparing it to the mass of steam that was injected. Steam displaces in front of it a large portion of the water and contaminants initially in the pore space, and because steam has a much lower density than water, it displaces a greater mass of water from the pores than its own mass. Thus, during the initial stages of a steam flood, more mass will be collected in the effluent than was injected as steam, and the production of a greater mass of effluent than was injected as steam confirms that a steam front was established.

Analytical Methods. Soil samples were analyzed by EPA SW-846 Method 8260B for volatile constituents, and by Method 8081 for organochlorine pesticides. Water samples were analyzed by a modification to Method 8270 that was developed by the analytical laboratory, Del Mar Analytical, of San Diego, CA. This method, designated Method 3520C/8270C, was developed to allow quantitation of chlorobenzene and DDT, DDD, and DDE simultaneously.

Experimental Results

Density as a Function of Temperature. Density of the DNAPL samples from each of the wells as a function of temperature in the range of 10°C to 90°C are shown in Table 1 and Figure 2. For comparison, the density of water as a function of temperature is also shown in Figure 2. The density of each of the DNAPL samples is similar. The differences between them are no more than about 0.007 gm/cm³ at any temperature. All the DNAPL samples show a similar decrease in density as the temperature increases of 0.001 gm/cm³/°C.

Analysis of the DNAPL done during previous investigations has shown that the DNAPL consists almost entirely of chlorobenzene and DDT. The physical properties of both chlorobenzene and the Montrose DNAPL (as reported in Hargis + Associates, H+A, 1999) are listed in Table 2. The specific gravity of chlorobenzene reported in literature is 1.1066 at 4°C (Verschueren, 1983), which is significantly less than the density of the Montrose DNAPL (H+A,

1999). There are an abundance of commercial fact sheets and articles available on DDT, and many of these list physical properties. However, there are considerable discrepancies between the various sources of information. Densities for DDT ranged from 1.016 to 1.55 gm/cm³ at 20°C in the references observed (a complete search of available information was not performed). Due to the lack of reliable literature information on the density of DDT, it cannot be determined if the densities measured in this experiment are what would be expected based on the densities of the pure components.

When the density of the DNAPL is compared to that of pure water, it can be seen that the buoyancy of the DNAPL increases as the temperature increases, although the change is relatively minor. Specifically, at 10°C the ratio of the density of the DNAPL to that of water ranges from 1.2284 to 1.2427. At 50°C, the ratio will range from 1.2023 to 1.2124, and at 90°C the ratios will range from 1.1878 to 1.1986. Thus, the data indicate that increasing the temperature of the subsurface will slightly increase the buoyancy of the DNAPL, or decrease the tendency of the DNAPL to move downward below the water table.

Viscosity as a Function of Temperature. Viscosity of the DNAPL samples as a function of temperature is shown in Table 3 and Figure 3. Viscosity measurements were only done at temperatures up to 60°C. At higher temperatures, the viscosity was outside of the range that can be measured with this apparatus. It would be expected that the viscosity would be leveling off at these temperatures, as is indicated in Figure 3, and would not decrease significantly at higher temperatures. Figure 3 shows that the viscosity of the site DNAPL decreases by approximately a factor of two as the temperature increases from 10°C to 60°C, thus doubling its mobility. The literature-reported viscosity of chlorobenzene is given in Table 2 as 0.753 at 25°C (Davis, 1998). DDT is normally a solid at these temperatures.

Surface and Interfacial Tension as a Function of Temperature. Table 4 and Figures 4A to 4D present the surface tension of the DNAPL samples, the surface tension of Montrose groundwater, and the interfacial tension between the groundwater and DNAPL. Initially an attempt was made to use water that had been treated by reverse osmosis (RO) for the interfacial tension measurements. However, when RO water was added to a DNAPL sample from well UBT-2, the water and DNAPL did not separate. The DNAPL adhered to the ring of the tensiometer, creating a thin layer of NAPL on top of the water which remained connected to the NAPL in the bottom through the ring. A picture of this is shown in Figure 5. Thus, site groundwater obtained from monitoring well MW-2 was used for all further measurements. It was noted in all interfacial tension measurements that a thin layer of the NAPL was always present on top of the water. Consequently, after making an interfacial tension measurement coming up through the interface, an additional measurement could not be made by going down through the interface. Thus, the surface tension data reported are an average of three measurements, but only one interfacial tension data point was obtained at each temperature.

The measured surface tension of the groundwater was always less than that of pure water, which is likely due to the presence of dissolved organics in the water. The surface tension also varied somewhat between samples of the groundwater. The surface tension of the groundwater decreased essentially linearly ($r = -0.996$ to -0.9998) with increasing temperature, as is expected. However, the rate of decrease with increasing temperature was slightly smaller than that of pure water, ranging from 0.11 to 0.15 dynes/cm/°C.

The surface tension of the four DNAPL samples was fairly consistent, with the largest differences between samples being approximately 1.5 dynes/cm. The surface tensions measured

at 10°C of approximately 35 dynes/cm are similar to published values of surface tension of 36.0 dynes/cm (see Table 2). The data shows that the surface tension of the DNAPL decreases linearly ($r=-0.97$ to -0.99996) as a function of temperature, with a slope of approximately 1 dyne/cm/°C.

The interfacial tension for all the DNAPL samples is approximately 11.0 - 11.5 dynes/cm. These values do not compare favorably with published values for the interfacial tension of chlorobenzene, which is 37 dynes/cm at 20°C (see Table 2). Also, it was found that the interfacial tension values for the DNAPL do not change significantly with temperature. Harrold et al. (2003) noted that waste samples of trichloroethylene (TCE) and tetrachloroethylene (PCE) obtained from field sites had different interfacial tension than reagent grade samples of the solvents. The majority of their data showed increases in interfacial tension when the solvent had been in contact with soils; only one soil type caused a decrease in interfacial tension of the solvents. Johnson and John (1999) found that dissolved humic substances of natural organic matter can decrease the interfacial tension of reagent grade PCE. Their data appears to show an exponential decrease in interfacial tension as a function of dissolved humic substances concentration. Thus, it is possible that passage through the soil caused the decrease in interfacial tension of the samples measured here. However, the interfacial tension measured here is substantially lower than what would be anticipated based on the interfacial tensions measured for the TCE and PCE samples exposed to soils or dissolved humic substances, which only went as low as about 30 dynes/cm.

Interfacial tension influences entry pressures and trapping potential for NAPL in the subsurface. The low interfacial tension of the DNAPL samples from this site indicate that it can more readily penetrate small pore throats than reagent grade chlorobenzene. However, the lack of a change in interfacial tension with temperature indicates that it would not have a greater ability to move into smaller pores during thermal remediation.

Steam Injection Column Experiments. A summary of the results of the steam injection column experiments is given in Table 5. A detailed description of the results of each of the experiments is given below.

Column #1. The first column experiment was performed with soil from soil boring SSB-6, from the 88 foot depth. The temperature history during this steam injection is shown in Figure 6. The graph shows that the temperature within the column reached approximately 115°C for a brief period early in the steam injection, and then decreased to approximately 105°C for the remainder of the steam injection. A mass balance on the water in the column shows that the column retained a water content of 20 to 40 mls throughout the steam injection (was not de-saturated). The initial and final soil concentrations of chlorobenzene, dichlorobenzenes (DCBs), and DDT are given in Table 6, and shown in Figure 7. Where there is a blank in the table, the constituent was not detected. Reporting limits for each of the constituents are given in Appendix A.

The three steamed soil samples that were analyzed showed similar results, indicating fairly uniform treatment of the soil throughout the column, with chlorobenzene concentrations ranging from 0.0061 to 0.048 mg/kg. Therefore, soil concentrations of chlorobenzene were reduced by approximately 99.97 percent. A small amount of 1,4-DCB was detected in the initial soil; it appears that the final soil concentrations of DCB were reduced by 90 percent. Both 2,4'-DDT and 4,4'-DDT were detected in the initial soil sample, at total DDT concentrations of more than three times the initial concentration of chlorobenzene. The steamed soil concentrations of

DDT were reduced to approximately one third of the initial soil concentrations. Small concentrations of 4,4'-DDD were also detected in the steamed soil. Figure 7 illustrates the dramatic reductions in chlorobenzene concentrations that were achieved, while only reducing DDT concentrations by a small amount.

Table 7 gives the water concentrations for Column #1, and these concentrations are shown graphically in Figure 8. The initial leachate concentration of chlorobenzene was found to be 2.9 mg/l, which is far less than the solubility of chlorobenzene of 490 mg/l at 25°C (Table 2). Small amounts of 2,4'-DDT and 4,4'-DDD were also found in the initial leachate sample. None of these compounds were detected in the final leachate (reporting limit of 0.10 mg/l). The effluent samples collected during the steam injection showed chlorobenzene only present in the effluent for the first two pore volumes of steam injected. The highest concentration in the effluent, as expected, was during the injection of the second pore volume of steam. After that time, the effluent concentrations of chlorobenzene decreased significantly. During the injection of the third and fourth pore volumes of steam, 2,4'-DDT, 4,4'-DDD, and 4,4'-DDT showed up in the effluent.

Small amounts of naphthalene (0.003 - 0.004 mg/kg) and m,p-xylene (0.0014 - 0.0017 mg/kg) were detected in the steamed soil. This was believed to be a carryover of contamination from previous treatability studies carried out with creosote contaminated soils and performed using the same equipment. Laboratory control samples for Method 8270C showed low recoveries of chlorobenzene (32 - 33 percent, while the acceptance criteria was 70 - 130 percent). Soil sample results for 2,4'-DDT and 4,4'-DDT were qualified due to co-elution of 2,4'-DDT and 2,4'-DDD in calibration samples. Chlorobenzene results in soil samples were not affected by quality control issues. The data quality is adequate for its intended use.

Column #2. The second column experiment was performed using soil from borehole SSB-6, from the 77 foot depth. The temperature history is shown in Figure 9. This figure shows that the temperature in the soil was maintained at approximately 105°C throughout the steam injection, with short-duration increases in the temperature occurring during pressure cycling. The initial and final soil sample results are given in Table 8, and shown graphically in Figure 10. As in Column #1, chlorobenzene concentrations were decreased by more than 99 percent by the injection of approximately four pore volumes of steam. Final soil chlorobenzene concentrations ranged from 0.11 to 0.64 mg/kg. A small amount of 1,4-DCB was detected in the initial soil, and its concentration was reduced by 99 percent in the steamed soil. Table 8 shows that the total DDT concentration in the steamed soil was three times the concentration in the initial soil. It is likely that this is due to the initial single soil sample not being representative of the DDT concentration of the soil actually packed into the column. Further evidence that the initial soil sample was not representative of the soil packed in the column is that the calculated mass of chlorobenzene recovered from the column was considerably greater than the mass calculated to be in the column initially based on the initial soil chlorobenzene concentration. The reductions detected in chlorobenzene and DCB concentrations, and increases detected in DDT concentrations, are illustrated in Figure 10.

Table 9 presents the water concentrations for Column #2. The initial soil sample showed a significant concentration of chlorobenzene (42 mg/l) which is approximately 10 percent of its solubility. Only 4,4'-DDT was detected in the final leachate sample at a relatively small concentration. Effluent sample #1 showed a surprisingly high concentration of chlorobenzene of 870 mg/l, which is approximately twice the solubility of chlorobenzene at ambient temperatures.

This likely indicates that some NAPL exited the column during the injection of the first pore volume of steam. Chlorobenzene concentrations drop off consistently during the injection of the second through fourth pore volumes of steam. Again, DDT showed up in the effluent during the injection of the third and fourth pore volumes of steam injection.

A small amount (0.001 mg/kg) of m,p-xylene was found in the steamed soil, which was likely due to contamination from creosote-contaminated soils that were tested in the same equipment previously. The chlorobenzene results for all of the aqueous samples for this experiment were qualified due to chlorobenzene being detected in the method blank at 0.0244 mg/l, and at 0.123 mg/l (246 percent recovery) in the Laboratory Control sample. There were no quality control issues with the soil sample results. The data can be used for its intended purpose.

Column #3. The third column experiment was performed with soil from boring PSB-4, from a depth of 88 feet below ground surface. The temperature history for this steam injection is shown in Figure 12. It can be seen that the maximum temperature in the soil column during this steam injection was approximately 120°C. It can also be seen that the temperature at the top of the column was higher than the temperature measured at the steam generator throughout the steam injection. This could indicate that there was some buildup of pressure within the column during the experiment, or that the heater tape was set too high. A water balance on the amount of water within the column during the steam injection showed that more water was collected than injected, indicating that a steam front formed in the column. Thus, it appears that excessive pressure did not build up during the experiment, and the observed high temperature at the top of the column thus had no effect on the experimental results. Initial and steamed soil concentrations are given in Table 10. Chlorobenzene concentrations in the soil were reduced by greater than 99.9 percent by the steam injection. DCB was not detected in the initial soil, however, small amounts (less than 1 mg/kg) were detected in the steamed soil. Initial soil DDT concentrations were greater than the chlorobenzene concentrations, and were reduced by approximately 50 percent by the steam injection. As observed in other columns, 4,4'-DDD was not detected in the initial soil, but was found in the steamed soil. These results are illustrated in Figure 13.

Table 11 presents the water concentrations for Column #3. The initial leachate contained 26 mg/l of chlorobenzene, which is only about five percent of its solubility limit. Only chlorobenzene was detected in the final leachate sample at a concentration of 18 mg/l, which is similar to the initial leachate concentration. The first two effluent samples contained only chlorobenzene and, as expected, the second sample contained a higher concentration of chlorobenzene. The concentration in this sample is more than an order of magnitude greater than the solubility of chlorobenzene, indicating that vaporization likely was the important removal mechanism for chlorobenzene. Only Effluent sample #3 contained DDT. These concentrations are illustrated in Figure 14, and show that chlorobenzene concentrations in the effluent were still high at the time the steam injection was terminated.

The trip blank associated with this set of analytical samples showed a chlorobenzene concentration of 0.29 mg/l. The laboratory control samples for Method 8270C showed somewhat low recoveries for chlorobenzene (48 – 49 percent). The method blank for Method 8081 contained a small concentration of DDT (0.00937 mg/kg), however, concentrations detected in soil samples were more than 10 times this concentration. Several laboratory control samples for Method 8081 for DDT were somewhat above the acceptance criteria (136 – 170 percent). The chlorobenzene concentrations in the soil were not effected by quality control

issues, thus, the data may be used for its intended purpose.

Column #3 Duplicate. A second steam injection experiment was performed using soil from the same borehole and depth interval as Column #3. The temperature history for this steam injection, shown in Figure 15, is similar to that of Column #3, with temperatures in the top of the column higher than at the steam generator, and temperatures in the soil of approximately 120°C. The water balance again indicates that a steam zone was formed in the column. Soil concentrations are given in Table 12, and show that the initial soil chlorobenzene concentration for this duplicate experiment was about twice that of the original Column 3 experiment. Steamed soil concentrations of chlorobenzene were again reduced by approximately 99.9 percent, with final concentrations ranging from 1.6 to 9.3 mg/kg. These steamed soil concentrations of chlorobenzene were about five times greater than found in the original Column #3. Although DCB was not detected in the initial soil, steamed soil samples showed small concentrations, reaching as high as 1.3 mg/kg. DDT concentrations in the initial soil were very similar to that in Column #3, with concentrations in the steamed soil reduced by about 18 percent, which is a smaller reduction in DDT concentrations than observed in previous experiments. The three orders of magnitude reduction in chlorobenzene concentrations is shown in Figure 16.

Aqueous concentrations for Column #3 duplicate are shown in Table 13 and Figure 17. Only chlorobenzene was detected in the initial leachate sample, and its concentration was approximately one tenth of its solubility. The chlorobenzene concentration in the final leachate sample was reduced significantly, and a small amount of 4,4'-DDT was detected. 4,4'-DDT was also detected in Effluent samples #1, #3, and #4, and 2,4'-DDT was detected in Effluent sample #4.

The trip blank associated with this set of analytical samples showed a chlorobenzene concentration of 0.45 mg/l, and the laboratory blank showed a concentration of 0.015 mg/l. Chlorobenzene concentrations detected in laboratory control samples for Method 8270C were low (57 percent recovery). m,p-Xylene was detected at 0.13 mg/kg in one of the steamed soil samples, and was also detected in the laboratory control sample at concentrations above the acceptance criteria (along with other fuel components). Chlorobenzene concentrations in soil were not effected by quality control issues. The data can be used for its intended purpose.

Column #4. The fourth column experiment was performed using soil from borehole PSB-15 at the 75 foot depth. The temperature history for this column experiment is shown in Figure 18. The figure shows that the temperature within the soil column remained at approximately 100°C or slightly below throughout the steam injection. Pressure cycling raised the temperature to approximately 110°C for short periods. Table 14 and Figure 19 present the soil concentration data for Column #4. Initial soils used for Columns #4 and #5 were spiked using DNAPL collected from one of the wells on site, because the initial soil results for chlorobenzene had been low compared to concentrations found in field soils during the DNAPL Reconnaissance Investigation. For this experiment, 14.5 ml of DNAPL collected from site well UBT-1 was added to 511.85 gms of soil. The soil and DNAPL were quickly mixed in a bowl, and then packed in the column. Thus, the initial soil concentrations of chlorobenzene and DDT for this experiment were 25,000 mg/kg and 26,800 mg/kg, respectively, which are significantly higher than the initial soil concentrations in the previously tested columns. The results for the steamed soil thus are also higher than in the other tested columns; however, the recovered percentage of chlorobenzene remained in the same range, at 99.9 percent. Steamed soil

concentrations of chlorobenzene were approximately 35 mg/kg, and DCB concentrations were less than 1 mg/kg. Concentrations of DDT in the steamed soil were generally one third less than in the initial soil.

Table 15 and Figure 20 show the aqueous concentrations for Column #4. Extremely large concentrations of chlorobenzene and DDT - well above the solubility limits of these compounds - were found in the initial leachate sample. This indicates that some DNAPL drained from the sample along with the leachate water. Surprisingly, no chlorobenzene or DDT was detected in the final leachate sample. Results from the four effluent samples are similar to what has been found in other experiments, where the chlorobenzene concentration peaks in the second pore volume of water injected, and DDT compounds are found in Effluent samples #3 and #4, as the chlorobenzene concentrations are declining.

Chlorobenzene concentrations in laboratory control samples for Method 8270C were below acceptance limits (42 – 43 percent). The method blank and laboratory control samples for DDT by Method 8081 exceeded method acceptance criteria (121 – 832 percent). This was caused by high concentrations in the samples that contaminated the batch quality control samples during the extraction process. Chlorobenzene concentrations in soil were not affected by quality control issues. The data quality is adequate for its intended purpose.

Column #5. The fifth column experiment was performed using soil from borehole SSB-2 from a depth of 86.5 feet below ground surface. For this experiment, 22 ml of DNAPL from site well UBT-1 was added to 890 gms of soil, and then a portion of this was packed into the column. The temperature history for this experiment is shown in Figure 21. The temperature in the top of the column (approximately 125 to 130°C) was greater than the temperature at the steam generator (120°C) throughout the steam injection, and the temperature in the middle of the column (approximately 123°C, then declining to 115°C) was also greater than that at the steam generator for the first half of the experiment. The temperature on the effluent line remained relatively low (90°C, then declining to 65°C) compared to that achieved during other experiments. The first effluent sample weight was 121.7 gms, which is greater than the 100 gms of steam that were injected during that time period. This indicates that a steam zone was likely being formed during the early portion of the steam injection. However, the weights of next three effluent samples were 77.3, 87.7, and 86.0 gms, respectively, indicating that the injected steam had condensed in the column. These data indicate that this column experiment was actually a hot water flood rather than a steam flood. This is discussed in more detail below.

Table 16 and Figure 22 show the soil concentrations for Column #5. These show that despite the lack of success with a steam flood in this experiment, soil concentrations of chlorobenzene were still reduced by more than 90 percent. However, the remaining soil concentrations, which ranged from 170 to 260 mg/kg, are substantially higher than the steamed soil concentrations in any of the previous experiments. The data also show that DDT concentrations were higher in the steamed soil than in the initial soil, indicating that the sample of the initial soil was likely not representative of the DDT concentrations of the soil packed into the column.

Table 17 and Figure 23 show the aqueous phase concentrations for Column #5. The initial leachate sample had a high chlorobenzene concentration of 110 mg/kg, approaching the solubility limit for chlorobenzene. However, the absence of DDT in this initial leachate sample indicates that it is unlikely that DNAPL eluted with this leachate sample. The final leachate sample shows a chlorobenzene concentration of 65 mg/kg, and high concentrations of DDT.

These high concentrations of chlorobenzene and DDT in the effluent samples indicate that DNAPL was likely displaced from the column throughout the hot water flood.

The trip blank associated with this set of samples showed chlorobenzene contamination at 0.38 mg/l. High levels of contamination in the samples caused contamination of the laboratory blanks during the extraction process. Soil samples analyzed by Method 8081 may be biased high for DDT due to this carryover. Volatilization losses of chlorobenzene during the volume reduction phase of the extraction process caused low laboratory control sample recoveries for chlorobenzene by Method 8270C. Chlorobenzene concentrations in soil samples were not effected by quality control issues, thus the data can be used for its intended use.

Discussion

Physical properties of the DNAPL. The physical properties of a non-aqueous phase liquid (NAPL) largely determine its fate and transport in the subsurface. The high density of the chlorobenzene/DDT DNAPL at the former Montrose plant property has allowed it to move significant distances below the water table, and its low interfacial tension has allowed it to move into the relatively fine-grained Bellflower Aquitard. Movement into the relatively low permeability aquitard was most-likely aided by the low to moderate viscosity of the DNAPL. The data collected here documenting the effect of temperature on the DNAPL density, viscosity, and surface and interfacial tension show that the effects of increasing temperature in a thermal remediation such as steam injection or electrical resistance heating would have only minor effects on the physical properties of the DNAPL. The DNAPL would become slightly more buoyant as the temperature is increased, decreasing the density gradient which creates downward migration, but the DNAPL would not become lighter than water. Based on the experimental data, the interfacial tension of the DNAPL and site groundwater essentially does not change with temperature, so the DNAPL will not move into finer-grained units than it is already in. Trapping of residual DNAPL below the water table will not be significantly affected. The decrease in surface tension of the DNAPL may allow some of the DNAPL trapped in the vadose zone to be mobilized, thus draining downward. However, it would for the most part be moving into areas that are already contaminated, and as the temperatures continue to increase, vaporization of the chlorobenzene would dominate. The vapors would remain in the vadose zone and could be collected. The lower viscosity at higher temperatures means that the DNAPL would be able to flow more readily as the temperature is increased, but this alone would not create a gradient to make the DNAPL move.

Although prior studies have found that the Montrose DNAPL is comprised mostly of chlorobenzene and DDT, the measured physical properties of the DNAPL from the field differed from that of either of the pure chemicals. The results obtained here are very similar to results reported in Hargis + Associates (1999). The DNAPL was found to have a greater density and a higher viscosity than pure chlorobenzene. Although the surface tension of the DNAPL was similar to that of chlorobenzene, the interfacial tension was substantially lower. It cannot be determined what affect the presence of the DDT dissolved in the chlorobenzene might have on the physical properties of the DNAPL because of the large discrepancies in published properties for DDT. Besides the discrepancies in the densities for DDT noted previously, there are also large differences in the published values of DDT solubility and boiling point (Pontolillo and

Eganhouse, 2001). These discrepancies make it difficult to determine how changes in the composition of the DNAPL would affect its movement.

One-dimensional steam injection experiments. Although the initial soil concentrations of chlorobenzene for the steam injections varied over almost three orders of magnitude, the percent reduction in chlorobenzene concentration remained fairly constant. Thus, the steamed soil concentrations varied over approximately four orders of magnitude. Steamed soil concentrations of chlorobenzene are shown as a function of the initial soil concentration in Figure 24, and it can be seen that there appears to be a linear relationship between them ($r=0.997$). The one exception to this appears to be for Column #2, which had the lowest initial soil concentration of chlorobenzene, but had a higher steamed soil concentration than Column #1. Since the two experimental runs had very similar temperature histories, the higher residual chlorobenzene concentration in Column #2 may be due to the higher silt content of the soil. Most of the contaminated soils appear to be mostly fine-grained sand, with some amount of silt, from which it is easier to recover chlorobenzene due to greater steam contact, and due to the fact that the smaller surface areas of sandy soils allows less adsorption of organic chemicals, as compared with silt or clay.

The soil used for Column #1 contained approximately five percent shell fragments. The presence of the shell fragments did not appear to affect the recovery of chlorobenzene, and the heat did not appear to adversely affect the properties of the shells and soil.

The data on DDT recovery is less straightforward to evaluate. The greater concentration of DDT in the steamed soil (post-steam versus pre-steam) of Columns #2 and #5 can be explained by a non-representative initial soil sample (Column #2), or by carryover problems in the laboratory (Column #5). The fact that more chlorobenzene was recovered in the effluent for Column #2 than was calculated to be in the column initially based on the initial soil sample results demonstrates that the initial soil sample was not representative of the soil that was packed in the column. Because the purpose of these experiments was to evaluate the ability of thermal remediation for chlorobenzene recovery, the lack of consistency in the soil DDT concentrations does not effect the conclusions made based on these experiments.

It is interesting that DDT was found in some of the effluent samples in relatively small amounts. For Columns #1, 2, 3, and 4, DDT was found in Effluent samples #3 and/or #4. By this time in the experiment, the bulk of the chlorobenzene appears to have already been recovered from the soil, as chlorobenzene concentrations in the latter two effluent samples are always smaller than in the second effluent sample. These experiments were all successful steam injections, so flow of liquid chlorobenzene from the column, carrying DDT with it, would not be expected during this portion of the experiment. For Columns #1 and 2, the maximum temperature during the steam injection was approximately 105°C, while for Columns #3 and 4 the maximum temperature was approximately 120°C. Published values of the melting point of DDT found in one source are 109°C for 4,4'-DDT, 74°C for 2,4'-DDT, 109-110°C for 4,4'-DDD, and 76-78°C for 2,4-DDD (Agency for Toxic Substances and Disease Registry). Thus, in Columns #1 and 2, the melting point of 4,4'-DDT and 4,4'-DDD had not been reached, but these compounds were found in effluent samples. And although the melting point for 2,4'-DDD had been exceeded in all the experiments, it was not commonly found in the effluent until the later part of the experiment. Had DDT DNAPL formed, and if it was present at saturations greater than residual saturations, the vertical orientation of the column and the downward direction of flow would have allowed it to flow freely from the column during the steam injection. This does

not appear to have happened.

The most likely mechanism for the DDT to appear in the effluent samples appears to be vaporization and transport out of the column with the steam and chlorobenzene vapors. Although a complete search of the available literature was not performed, some of the available information on the physical properties of DDT was consulted in an effort to determine the boiling point and vapor pressure of DDT. Considerable variations are noted in the boiling points published in different sources, starting as low as 185°C. Some sources say that DDT decomposes before it boils, and list a boiling point for 4,4'-DDD of 350°C (Agency for Toxic Substances and Disease Registry). No boiling point data was available on 2,4'-DDT or 2,4'-DDD. Vapor pressure data that is available includes: 4,4'-DDT, 1.6×10^{-7} torr at 20°C; 4,4'-DDD, 1.35×10^{-6} torr at 25°C; 2,4'-DDT, 1.1×10^{-7} torr at 20°C; and 2,4'-DDD, 1.94×10^{-6} torr at 30°C. Despite these very low vapor pressures, it has been found that significant DDT volatilization from crop lands can occur (Nash and Beall, 1970; Spencer et. al, 1996). Thus, the most likely explanation for the DDT found in effluent samples from column experiments where steam zones were formed is vaporization from the soil with subsequent transport with the steam and chlorobenzene vapors.

4,4'-DDD was found in steamed soil samples from Columns #1, 2, 3, and 4, but was only detected in the initial soil sample for Column #4. Small amounts of 4,4'-DDD were also found in effluent samples for Columns #1, 2, 4, and 5. The lack of detection of 4,4'-DDD in initial soil samples was likely due to high detection limits (25 - 750 mg/kg) for the initial soil samples which masked the presence of this compound. 4,4'-DDD is known to be present in Montrose site soils. Hydrolysis data for 4,4'-DDD shows that it has a half-life of 5.9 days at 85°C and pH 7.22. Although these laboratory experiments were too short of time duration for significant hydrolysis to occur, hydrolysis of DDD during a thermal remediation may be significant (Ellington et. al, 1987).

Column #5. Although the temperature in Column #5 reached approximately 123°C, there is a variety of evidence to support the conclusion that this experiment amounted to a hot water flood of the soil rather than a steam flood. First, it was observed that the total amount of effluent collected during the steam injection was considerably less than the amount of water injected as steam. This is shown in Figure 25, a graph of the mass of effluent collected as a function of the mass of water injected as steam. This graph shows for columns 1 through 4, more effluent was collected than the amount of steam injected. This is due to the fact that the steam front will displace in front of it some of the water that was initially in the column. For Column #5, the first sample contained a greater mass of effluent than the amount of steam that was injected, indicating a steam zone was starting to form. However, each of the samples after that contained less mass than the amount of steam injected, which indicates that during this time the injected steam was condensing. The condensation of the steam could be caused by high pressures in the column, and indeed, it was noted during the experiment that during pressure cycling, the pressure increased much more quickly than it had during other experiments. Also, when the steam injection was complete and the steam injection line disconnected from the column, liquids were vented from the top of the column for about five seconds. This is another indication that there was significant pressure within the column. It is not known what caused the pressure buildup. Although partial plugging of the effluent line could have caused the observed pressure buildup, the blockage was not complete, as a significant amount of effluent was collected.

Observation of the concentrations in the effluent samples shows that all concentrations are higher in the first effluent sample than in the remaining samples, and that the DDT concentrations in all effluent samples were higher than in other experiments. Only for this experiment were there significant DDT concentrations in all effluent samples. This can likely be explained by the displacement of DNAPL from the column. DNAPL was observed in the container for the first effluent sample. Also, the chlorobenzene concentrations in each of the effluent samples are greater than the solubility of chlorobenzene at ambient temperatures. Data on the solubility of chlorobenzene as a function of temperature is not readily available. An attempt was made to find data on the solubility of DDT. A thorough search of the literature for solubility data on DDT and DDE was undertaken by Pontolillo and Eganhouse (2001). They performed a critical review of the information available and concluded that none of the data available is reliable either because the method used for making the measurement was inadequate, or the method was not documented adequately to determine if the measurement was reliable. The published original data reviewed by Pontolillo and Eganhouse (2001) generally reported solubility to range from 0.001 to 0.1 mg/l; however, one reported value was 6.22 mg/l. Based on the range of data that are generally reported on the solubility of DDT, the very high concentrations of DDT in the effluent samples from Column #5 indicate the presence of DNAPL in the sample.

Despite the fact that this column experiment was a hot water flood instead of a steam flood, there was a surprisingly high recovery of chlorobenzene, approximately 90 percent. This demonstrates that the chlorobenzene/DDT DNAPL is readily displaced by hot water. However, it must be kept in mind that the vertical orientation of the column and downward flow in this case likely enhanced the recovery of DNAPL from the column. The final chlorobenzene concentration in the soil in this column is considerably higher than in the steam injection columns, which shows the ability of the steam to significantly reduce the residual saturation of volatile organic compounds by vaporization.

Leachate analysis results. Leachate samples were obtained to estimate the concentration of chlorobenzene and DDT in equilibrium with water for both the initial soils and the post-steamed soils. A sample of the water used to produce the leachate samples was characterized to provide a baseline analysis and found to be free of chlorobenzene and DDT. Leachate samples were generated using an approximately 24 hour contact time between soils and water, although it is recognized that this time period may not be adequate for complete equilibrium of the water with the soil. Also, for final leachate samples, the water was usually added to the steamed soil while the soil was still hot, thus more solubilization of the contaminants may have occurred. Thus, the final leachate concentrations may represent a conservative concentration. Keeping these limitations in mind, the data still show that after thermal remediation, groundwater concentrations of chlorobenzene would likely be reduced. Figure 26 shows leachate concentrations as a function of soil concentration for both initial and steamed soil. Note that the initial leachate sample for Column #4 is not included in this figure, as results indicate that DNAPL was in the sample.

Although all of the initial leachate samples had detectable concentrations of chlorobenzene, chlorobenzene was not detected in three of the final leachate samples (reporting limits were 0.071 to 0.12 mg/l). This is consistent with prior research which showed that in aged soils, a significant portion of the adsorbed chlorobenzene is non-desorbable (Sharer et. al, 2003). Two of the other final leachate samples had very low concentrations of chlorobenzene. The final

leachate sample from Column #5 had a relatively high concentration of chlorobenzene, but also had the highest final ('steamed') soil chlorobenzene concentration due to the fact that it was a hot waterflood rather than a steam flood. It appears that groundwater concentrations of chlorobenzene would be reduced after thermal remediation, although there is not a linear relationship between chlorobenzene concentration in the soil and in the leachate, as would be expected if adsorption were fully reversible.

DDT was generally not found in the leachate samples. Small amounts of 2,4'-DDT and 4,4'-DDT were found in the initial leachate sample for Column #1, and small amounts were found in the final leachate sample for Columns #2 and #3 duplicate. Significant concentrations which are likely above the solubility of DDT were found in the final leachate sample for Column #5, which was a hot water flood rather than a steam flood. The high concentrations in the final leachate for Column #5 cannot readily be explained. In some cases it has been found that heating contaminated soils to steam temperatures has caused dissolution of contaminants into the aqueous phase, and the contaminants appear to remain in the aqueous phase after soils have returned to ambient temperatures. This may have occurred here.

Duplicate column experiments using soil from PSB-4, 88 ft. As called for in the treatability study work plan, duplicate steam injection experiments were performed using one of the soil samples. PSB-4, 88 ft was the logical choice of soil samples for the duplicate column because adequate sample was available to run two columns. Comparison of the results can be seen in Tables 10 through 13, which are shown graphically in Figures 12 to 17. Although DDT concentrations in the two initial soil samples were similar, the chlorobenzene concentrations differ by approximately 65 percent. This indicates some of the variability in soil concentrations from the same field location. The first of these columns reached a maximum temperature of approximately 130°C at the middle of the column, while the second reached 125°C. The duplicate column reached steam temperatures in the effluent line earlier in the experiment than did the first column, and although the same amount of steam was injected into both columns, approximately 40 ml more of effluent was collected from the second column than from the first. These are typical variations that occur between the steam injection experiments that are carried out in the laboratory. Factors such as the amount of air flow through the hood where the experiments are performed or differences in insulation wrapping of the column can cause the observed differences in the steam and column temperatures. The degree of sensitivity of the steamed soil concentrations to these variations is unknown. However, the differences in steamed soil concentrations for these two columns is greater than the expected variations in the analytical method (± 40 percent). The initial leachate concentrations and the steamed soil concentrations seem overall to be proportional to the initial soil concentrations. Small concentrations of DCB were detected in steamed soil samples from both columns. Overall, the two column experiments show the same trends in the effluent samples.

Comparison of Analytical Methods. The treatability study work plan called for the use of EPA Method 8260 to determine chlorobenzene and Method 8081 to determine DDT in soil samples, while Modified Method 8270C was called for to analyze for both chlorobenzene and DDT in the aqueous samples. Different methods were used for the different media because both chlorobenzene and DDT concentrations were desired, but there was not enough of the aqueous sample to perform two analyses. For the soil samples, plenty of soil was available and the lower detection limits afforded by Methods 8260 and 8081 were needed for the steamed soil samples. At the time the soil samples were collected in the field, a sample was also taken for analysis by

modified Method 8270. Comparing results from these methods, it was noted that the results from modified Method 8270 were always higher than the results from Method 8260. This could have been due to the longer storage time for the treatability study samples and their handling in the laboratory as columns are packed, or it could have been due to differences in the two different analytical methods. For Column #5, soil samples were submitted for analysis by all three methods. The results are summarized in Table 18. It can be seen that Method 8270C, for this set of samples, resulted in higher concentrations of chlorobenzene and DDT, consistent with observed differences between these methods.

Conclusions

Measurements of the physical properties of the chlorobenzene/DDT DNAPL indicate that there should be no adverse effects on the distribution of the DNAPL (e.g., enhanced downward migration, increased infiltration into fine-grained layers, enhanced migration of DDT, etc.) from raising the temperature of the subsurface. The relatively low viscosity of the DNAPL means that it is readily displaced as a liquid, and a hot water flood through soils which contain free phase DNAPL potentially could reduce chlorobenzene concentrations by as much as an order of magnitude. Some DDT likely would be recovered with the DNAPL. However, elevated concentrations of chlorobenzene and DDT would remain in the soil, with concentrations likely in the range of hundreds to thousands of milligrams per kilogram of soil. The main mechanism for DNAPL recovery during a hot water flood would be displacement, with viscosity reduction a secondary mechanism.

By creating a steam front in the soil, vaporization also becomes an important mechanism for the recovery of residual chlorobenzene left behind by the hot water flood, reducing residual chlorobenzene concentrations by an order of magnitude more than what can be achieved with a hot water flood. In addition, it is likely that small amounts of DDT could also be vaporized and removed in the vapor phase. Chlorobenzene removals during steam floods ranged from 99.04 to 99.97 percent, resulting in steamed soil concentrations ranging from 0.0026 to 36.7 mg/kg. These concentrations are likely below residual DNAPL saturation, and DNAPL was never observed in the steamed soil samples. A white precipitate believed to be DDT was observed in some steamed soil samples. Peak chlorobenzene concentrations were found in the second pore volume of effluent, however, some of the column experiments still had significant amount of chlorobenzene in the effluent at the end of the steam injection, indicating that lower final soil concentrations likely could have been achieved with additional steam injection.

The DNAPL Field Reconnaissance (Hargis + Associates, 2004) found that most of the chlorobenzene and DDT contamination is located in soils classified as poorly graded sands with little or no fines (SP), silty sands (SM), or inorganic silts and very fine sands with slight plasticity (ML). The thickness of these soil lenses ranged from 0.1 to 4 feet. Since the SP sands are likely the most permeable units of those containing DNAPL at the former Montrose plant property, they may be expected to accept and be swept well by steam. Because the less permeable layers are thin, they would likely be heated thoroughly by heat conduction where they do not accept steam directly. With the use of pressure cycling, performed by halting steam injection while continuing to extract liquids and vapors, vapors could be recovered from these lower permeability zones. Because remediation treatment times are much longer in the field (on the order of months) than in the laboratory (approximately 5 hours), a greater level of treatment

may be possible in the field than was achieved in the lab study. Thus, it can be concluded that steam injection would be effective for recovery of chlorobenzene DNAPL from the Montrose plant property soils. Most of the chlorobenzene DNAPL may be recovered with the initial pore volumes of steam injected. Pressure cycling would enhance the vaporization of chlorobenzene and its removal from the low permeability zones (Itamura and Udell, 1995).

In the steam floods, roughly 25 to 50% of the DDT was removed from the soil. When DDT was detected in effluent samples, it was usually in the third and fourth samples, after the peak of chlorobenzene was past. It is believed that the most likely mechanism for DDT removal is vaporization, although the boiling point and vapor pressure of DDT are not known. This is a distillation process, where the lower boiling compounds vaporize first, then the higher boiling compounds. Thus, it makes sense that after the lower boiling chlorobenzene is largely removed from the column, the DDT (which has a lower vapor pressure) would begin to distill off to a greater extent. The fact that the melting point of DDT was not reached in some of these experiments, but DDT was detected in the effluent, indicates that downward mobilization of DDT DNAPL was not likely the mechanism for DDT in effluent samples. Several of the column experiments reached temperatures above the melting point of DDT; however, the effluent from these columns did not contain significantly more DDT. Thus, it does not appear that liquid DDT exited the column.

There were some issues identified as part of the laboratory quality assurance checks, including carry-over, chlorobenzene volatilization losses, and variations in reported concentrations between analytical methods. However, soil results for chlorobenzene were not impacted, and the data is adequate for the purposes of this treatability study. The quality assurance problems of carryover and differences between the analytical methods would adversely affect efforts to do a mass balance on the amount of contaminants recovered from the soil and the concentrations found in the effluent samples, thus, mass balance results have not been presented. Carryover problems of DDT may have affected some soil sample results.

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10. Initial and final soil concentrations for steam injection Column #2.
11. Aqueous concentrations for steam injection Column #2.
12. Temperature history during steam injection for Column #3.
13. Initial and final soil concentrations for steam injection Column #3.
14. Aqueous concentrations for steam injection Column #3.
15. Temperature history during steam injection for Column #3 duplicate.
16. Initial and final soil concentrations for steam injection Column #3 duplicate.
17. Aqueous concentrations for steam injection Column #3 duplicate.
18. Temperature history during steam injection for Column #4.
19. Initial and final soil concentrations for steam injection Column #4.
20. Aqueous concentrations for steam injection Column #4.
21. Temperature history during steam injection for Column #5.
22. Initial and final soil concentrations for steam injection Column #5.

23. Aqueous concentrations for steam injection Column #5.
24. Comparison of initial and steamed soil chlorobenzene concentrations.
25. Weight of effluent collected as a function of the mass of steam injected for each of the column experiments.
26. Leachate concentrations as a function of soil concentration for initial and steamed soils.

Table 1. Density of Montrose DNAPL as a Function of Temperature (gm/cm ³)				
Temperature °C	UBT-1	UBT-2	UBT-3	UBE-1
10	1.234 ± 0.0002	1.239 ± 0.0002	1.228 ± 0.0004	1.233 ± 0.0001
20	1.224 ± 0.0001	1.229 ± 0.0002	1.217 ± 0.0001	1.222 ± 0.0005
30	1.214 ± 0.0002	1.218 ± 0.0005	1.209 ± 0.0003	1.211 ± 0.0007
40	1.202 ± 0.0002	1.209 ± 0.0005	1.199 ± 0.0002	1.200 ± 0.0005
50	1.194 ± 0.0001	1.198 ± 0.0002	1.188 ± 0.0002	1.190 ± 0.0004
60	1.186 ± 0.0001	1.188 ± 0.0005	1.178 ± 0.0003	1.181 ± 0.0007
70	1.174 ± 0.0001	1.177 ± 0.0008	1.167 ± 0.0004	1.171 ± 0.0007
80	1.163 ± 0.0002	1.165 ± 0.0023	1.157 ± 0.0003	1.160 ± 0.0005
90	1.154 ± 0.0002	1.157 ± 0.0002	1.146 ± 0.0004	1.150 ± 0.0002

Table 2. Physical Properties of Chlorobenzene and Montrose DNAPL		
	Chlorobenzene	Montrose DNAPL ^d
Specific Gravity or Density	1.1066 at 4°C ^a	1.241 - 1.252 gms/cm ³
Viscosity	0.753 cP at 25°C ^b	2.5 - 2.8 cP
Surface Tension	35.97 dynes/cm ^c	
Interfacial Tension	37.4 dynes/cm at 20°C ^c	13 - 15 dynes/cm
Solubility	490 mg/l at 25°C ^b	

a - Verschuereen, 1983

b - Davis, 1998

c - Mercer and Cohen, 1993

d - Hargis + Associates, 1999; all measurements at 22°C

Table 3. Viscosity of Montrose DNAPL as a Function of Temperature (centiPoise)				
Temperature °C	UBT-1	UBT-2	UBT-3	UBE-1
10	3.41 ± 0.042	3.4333 ± 0.041	3.4 ± 0.707	3.46 ± 0.424
20	2.80	2.81 ± 0.042	2.76 ± 0.042	2.78 ± 0.041
30	2.33 ± 0.041	2.32 ± 0.041	2.28 ± 0.041	2.33 ± 0.041
40	2.03 ± 0.041	2.01 ± 0.042	2.05	2.05
50	1.93 ± 0.041	1.8	1.86 ± 0.042	1.86 ± 0.042
60	1.93 ± 0.041	1.8	1.85	1.83 ± 0.041

Table 4. Surface and Interfacial Tension of Montrose DNAPL and Groundwater as a Function of Temperature (dynes/cm)				
Temperature °C		10	50	90
UBT-1	DNAPL Surface Tension	35.9 ± 0.07	32.3 ± 0.34	27.7 ± 0.13
	Groundwater Surface Tension	67.3 ± 0.33	61.3 ± 0.10	56.7 ± 0.27
	Interfacial Tension	11.4	11.1	11.4
UBT-2	DNAPL Surface Tension	35.6 ± 0.20	32.6 ± 0.13	27.6 ± 0.29
	Groundwater Surface Tension	67.2 ± 0.3	61.5 ± 0.26	55.4 ± 0.40
	Interfacial Tension	11.2	10.9	10.6
UBT-3	DNAPL Surface Tension	35.1 ± 0.15	31.0 ± 0.13	26.7 ± 0.07
	Groundwater Surface Tension	63.6 ± 0.40	59.7 ± 0.40	54.4 ± 0.39
	Interfacial Tension	11.1	11.3	11.6
UBE-1	DNAPL Surface Tension	34.5 ± 0.40	31.1 ± 0.34	26.8 ± 0.19
	Groundwater Surface Tension	67.2 ± 0.21	61.2 ± 0.26	56.4 ± 0.38
	Interfacial Tension	11.5	11.1	11.8

Table 5. Summary of Results of the One Dimensional Steam Injection Experiments for Montrose Soils

	Soil Type (from boring log)	Initial Chlorobenzene concentration mg/kg	Final Chlorobenzene concentration mg/kg	Initial DDT concentration mg/kg (Total)	Final average DDT concen- tration mg/kg (Total)	Percent Chlorobenzene Removal	Comments
Column #1 SSB-6 88 ft	Sand with silt, light olive gray, wet, weak, poorly sorted, fine- to coarse grained, ~ 5% shell fragments	85	0.026	300	102	99.97	Maximum temperature of ~105C
Column #2 SSB-6 77 ft	Silt, light yellowish brown, wet, very stiff, low plasticity, with sand interbeds	39	0.373	142	703.3	99.04	Maximum temperature of ~105C,
Column #3 PSB-4 88 ft	Sand with silt, wet, noncemented, well sorted, fine- grained, subrounded and Silt, sand with silt, olive, stiff, nonplastic to low plasticity, mottled	2,600	0.993	4,400	2,673	99.96	Maximum temperature of ~120C at middle of column
Column #3 duplicate PSB-4 88 ft	same as above	4,300	5.6	4,400	3,583	99.87	Same as above
Column #4 PSB-15 75 ft	Silty sand, sand and silt, weak, wet, well sorted, fine grained	25,000	36.7	26,800	19,776.7	99.85	Maximum temperature of ~100C
Column #5 SSB-2 86.5 ft	Sand, olive gray, wet, weak, fine to medium grained	3,200	210	13,100	15,567	93.44	Hot waterflood, maximum temperature of ~123C

Table 6. Soil concentration results for Column #1 (mg/kg)

SSB-6 88 ft	Chlorobenzene	1,2-Dichloobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Soil	85		0.910	100		200	300
Steamed Soil-Top	0.025	0.0014	0.011	36	10	76	122
Steamed Soil-Top-Duplicate	0.048		0.007	34		70	104
Steamed Soil-Bottom	0.0061			23	6.7	50	79.7

Table 7. Water concentration results for Column #1 (mg/l)

SSB-6 88 ft	Chlorobenzene	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Leachate	2.9	0.4		0.45	0.85
Final Leachate					
Effluent Sample #1	20				
Effluent Sample #2	25				
Effluent Sample #3	1.6	0.26	0.11	0.42	0.79
Effluent Sample #4	0.49	0.16	0.074	0.260	0.494

Table 8. Soil concentration results for Column #2 (mg/kg)

SSB-6 77 ft	Chlorobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Soil	39	0.6	32		110	142
Steamed Soil-Top	0.11	0.0016	97		410	507
Steamed Soil-Bottom	0.64		210	53	720	983
Steamed Soil-Bottom Duplicate	0.37		140		480	620

Table 9. Water concentrations for Column #2 (mg/l).

SSB-6 77 ft	Chlorobenzene	2,4'-DDD	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Leachate	42					
Final Leachate					0.31	0.31
Effluent Sample #1	870					
Effluent Sample #2	170					
Effluent Sample #3	25		4.8	1.0	7.4	13.2
Effluent Sample #4	9	0.600	4.0	1.0	6.1	11.7

Table 10. Soil concentrations for Column #3 (mg/kg)

PSB-4-88 ft	Chlorobenzene	1,2-Dichloobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Soil	2,600			1,300		3,100	4,400
Steamed Soil-Top	0.69		0.16	680	260	1,800	2,740
Steamed Soil-Top-Duplicate	0.59		0.24	640	250	1,800	2,690
Steamed Soil- Bottom	1.7	0.16	0.94	640	250	1,700	2,590

Table 11. Water concentrations for Column #3 (mg/l)

PSB-4-88 ft	Chlorobenzene	2,4'-DDT	4,4'-DDT	Total DDT
Initial Leachate	26			
Final Leachate	18			
Effluent Sample #1	100			
Effluent Sample #2	6,400			
Effluent Sample #3	250	10	15	25
Effluent Sample #4	150			

Table 12. Soil concentrations for Column #3 duplicate (mg/kg)

PSB-4-88 ft	Chlorobenzene	1,2-Dichloobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDT	Total DDT
Initial Soil	4,300			1,100	3,300	4,400
Steamed Soil-Top	1.6		0.3	1,000	3,100	4,100
Steamed Soil-Bottom	9.3	0.25	1.3	750	2,700	3,450
Steamed Soil- Bottom Duplicate	6.0		0.89	800	2,400	3,200

Table 13. Water concentrations for Column #3 duplicate (mg/l)

PSB-6 88 ft	Chlorobenzene	2,4'-DDT	4,4'-DDT	Total DDT
Initial Leachate	32			
Final Leachate	0.5		0.13	0.13
Effluent Sample #1	410		6.9	6.9
Effluent Sample #2	6,100			
Effluent Sample #3	320		6.2	6.2
Effluent Sample #4	97	3.1	7.2	10.3

Table 14. Soil concentrations for Column #4 (mg/kg)

PSB-15-75 ft	Chlorobenzene	1,4-Dichlorobenzene	2,4'-DDD	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Soil	25,000			9,000	2,800	15,000	26,800
Steamed Soil-Top	35	0.95		5,600	1,700	13,000	20,300
Steamed Soil- Top Duplicate	37	0.77	830	6,800	1,700	2,100	11,430
Steamed Soil- Bottom	38	0.87		5,700	2,100	14,000	21,800

Table 15. Water concentrations for Column #4 (mg/l)

PSB-15-75 ft	Chlorobenzene	2,4'-DDD	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Leachate	1,400	190	1,900	610	4,200	6,900
Final Leachate						
Effluent Sample #1	53					
Effluent Sample #2	6,000					
Effluent Sample #3	320		66	17	100	183
Effluent Sample #4	140	8.2	58	14	68	148.2

Table 16. Soil concentrations for Column #5 (mg/kg)

SSB-2 86.5 ft	Chlorobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDT	Total DDT
Initial Soil	3,200		3,300	9,800	13,100
Steamed Soil-Top	200	2	3,700	9,800	13,500
Steamed Soil- Top Duplicate	260	3	4,900	13,000	17,900
Steamed Soil- Bottom	170		4,300	11,000	15,300

Table 17. Water concentrations for Column #5 (mg/l)

SSB-2 86.5 ft	Chlorobenzene	2,4'-DDD	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Leachate	110					
Final Leachate	65		2.7	1.6	6.9	11.2
Effluent Sample #1	5,000	230	2,600	640	3,000	6,470
Effluent Sample #2	1,200		87	37	250	374
Effluent Sample #3	600		26	15	76	117
Effluent Sample #4	880		30		94	124

Table 18. Comparison of analytical results for EPA Methods 8260B and 8081 with Modified Method 8270C (mg/kg)

SSB-2 86.5 ft	Modified Method 8270C	Methods 8260/8081
Chlorobenzene	34,000	3,200
2,4'-DDT	12,000	3,300
4,4'-DDT	31,500	9,800

Figure 1. Experimental apparatus.

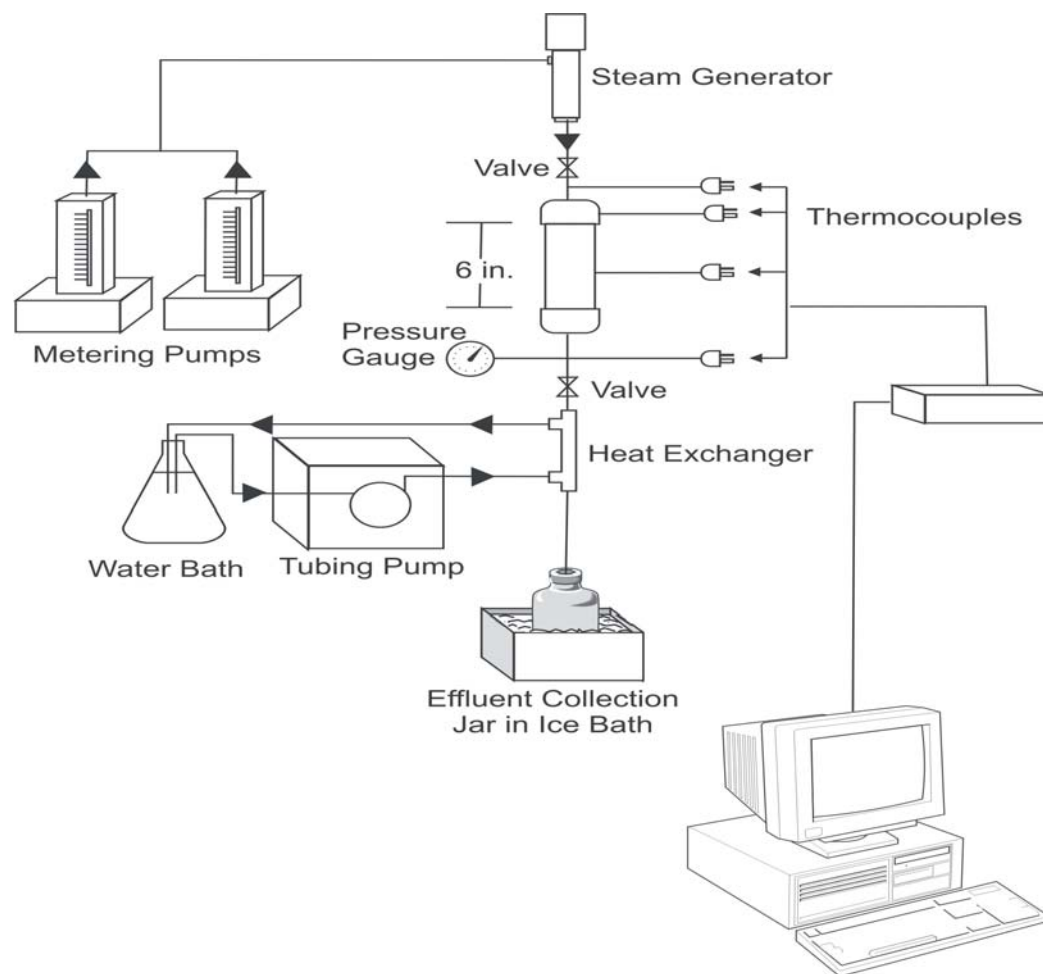


Figure 2. Density of Montrose DNAPL as a function of temperature.

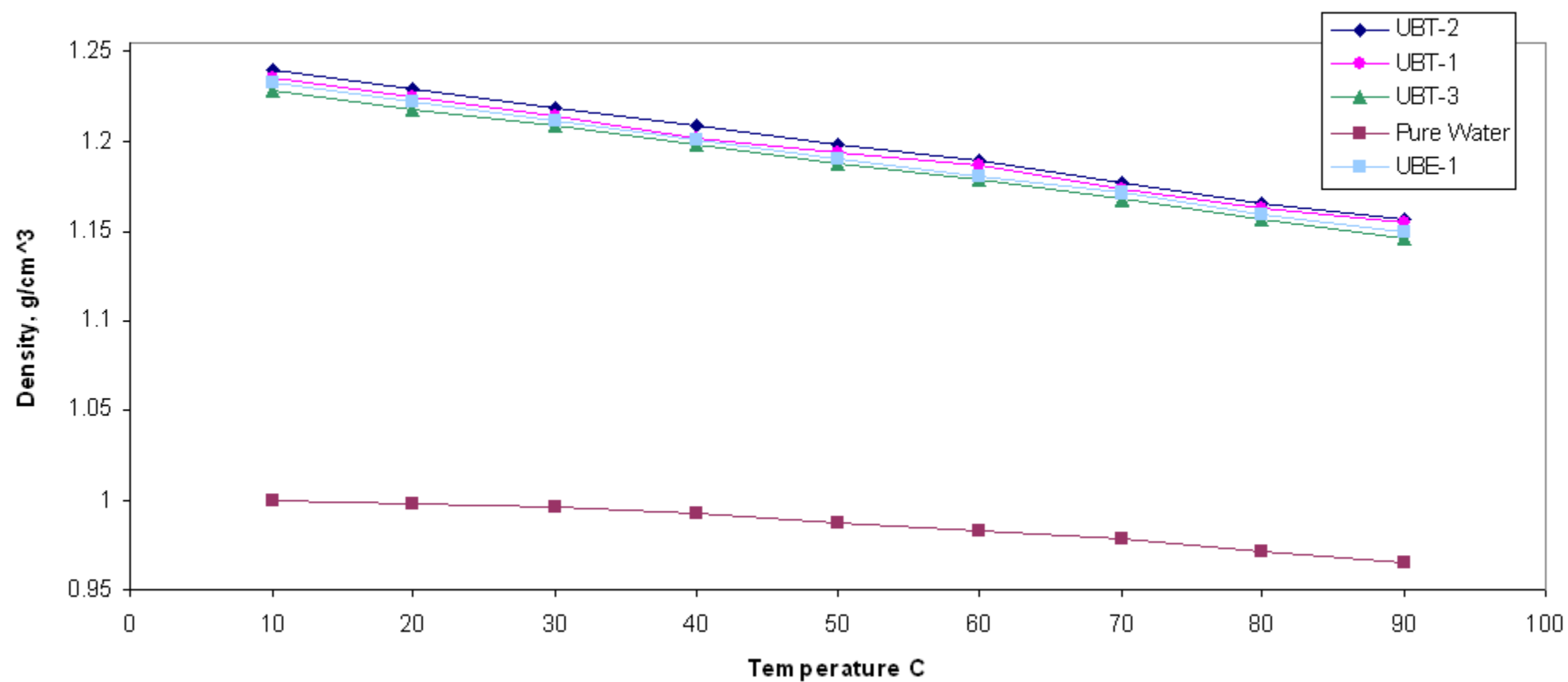


Figure 3. Viscosity of Montrose DNAPL as a function of temperature.

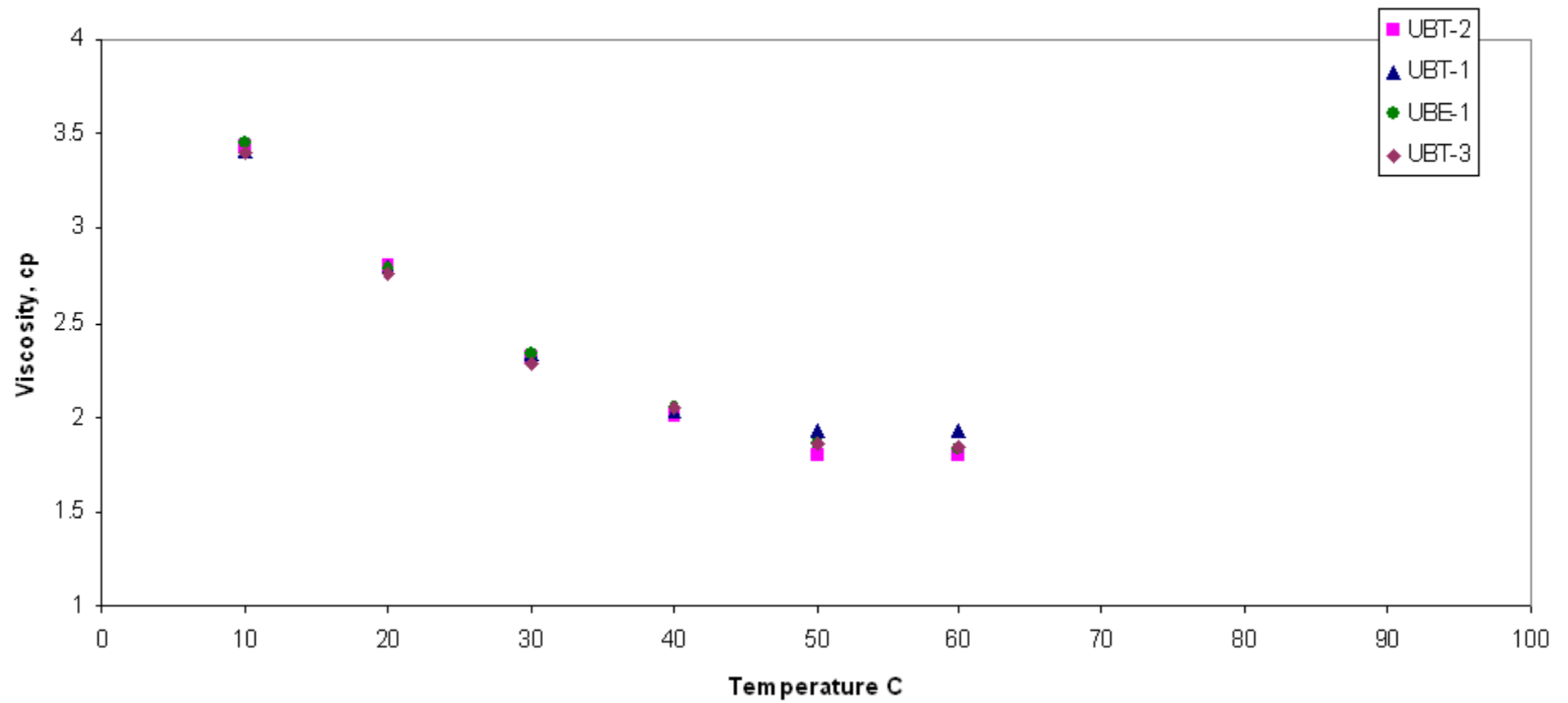


Figure 4A. Surface and interfacial tension of Montrose DNAPL and water as a function of temperature.

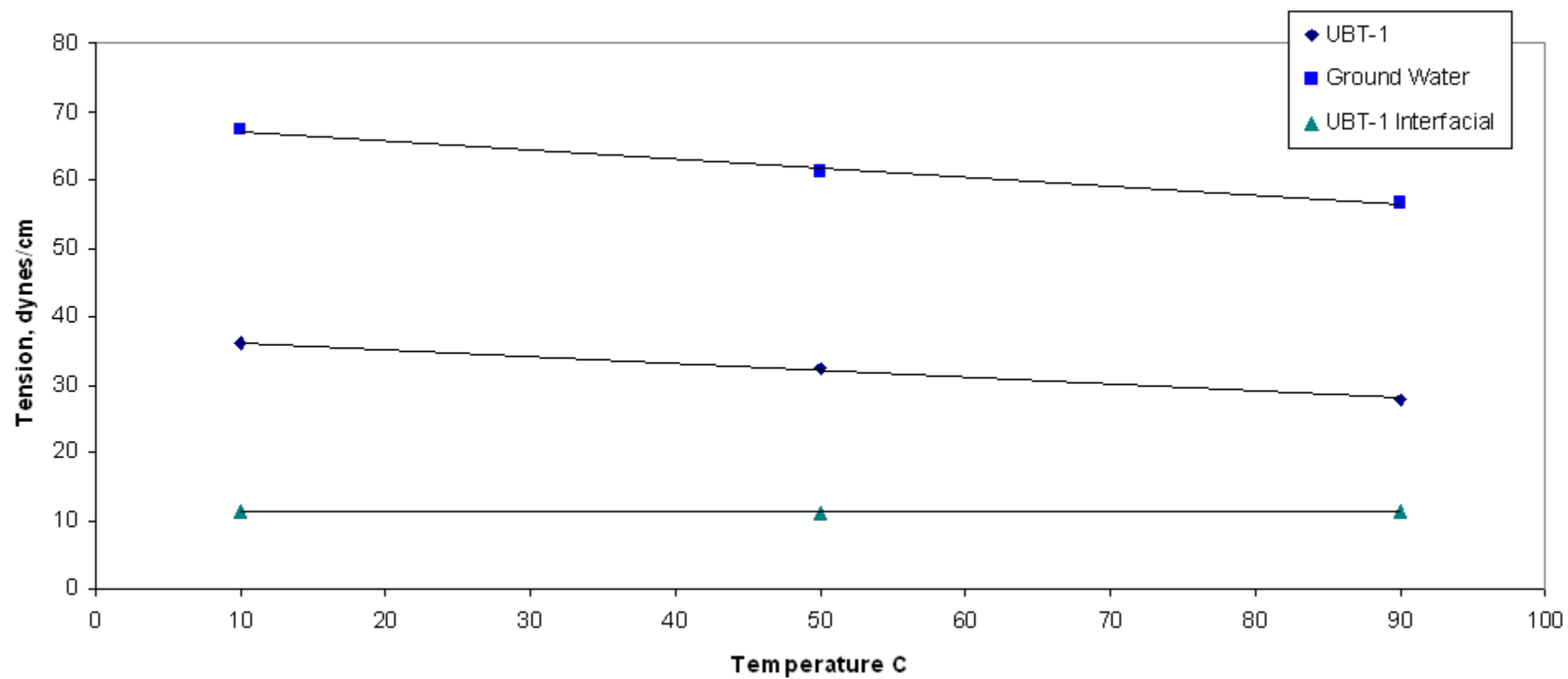


Figure 4B. Surface and interfacial tension of Montrose DNAPL and water as a function of temperature.

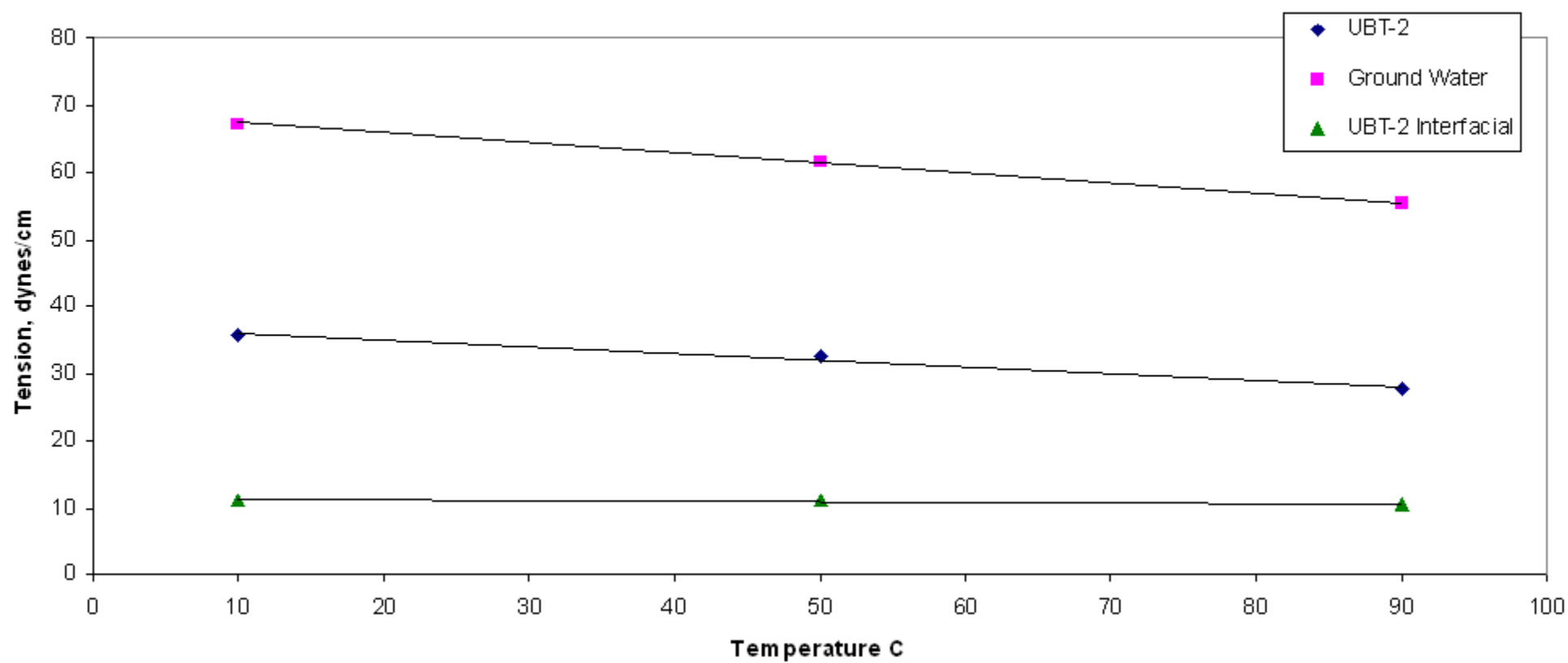


Figure 4C. Surface and interfacial tension of Montrose DNAPL and water as a function of temperature

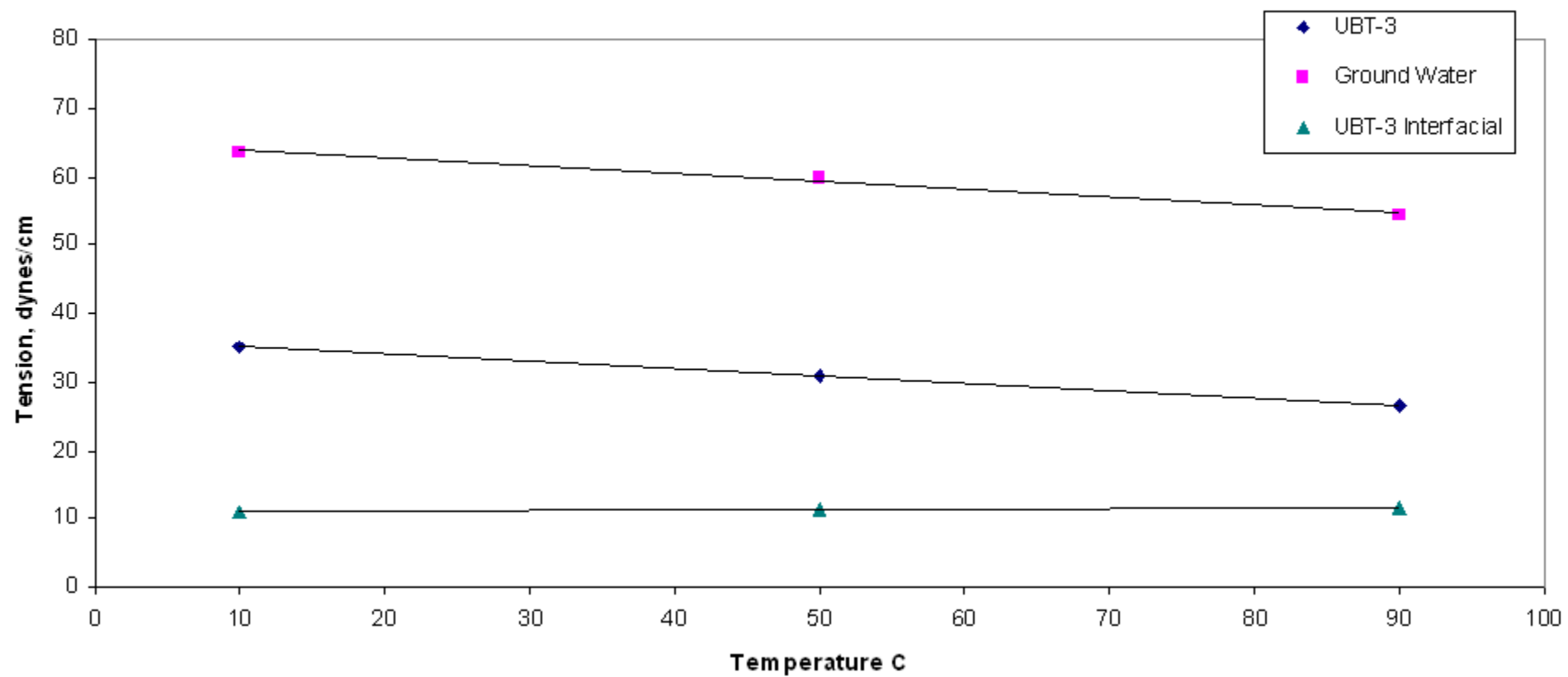


Figure 4D. Surface and interfacial tension of Montrose DNAPL and water as a function of temperature.

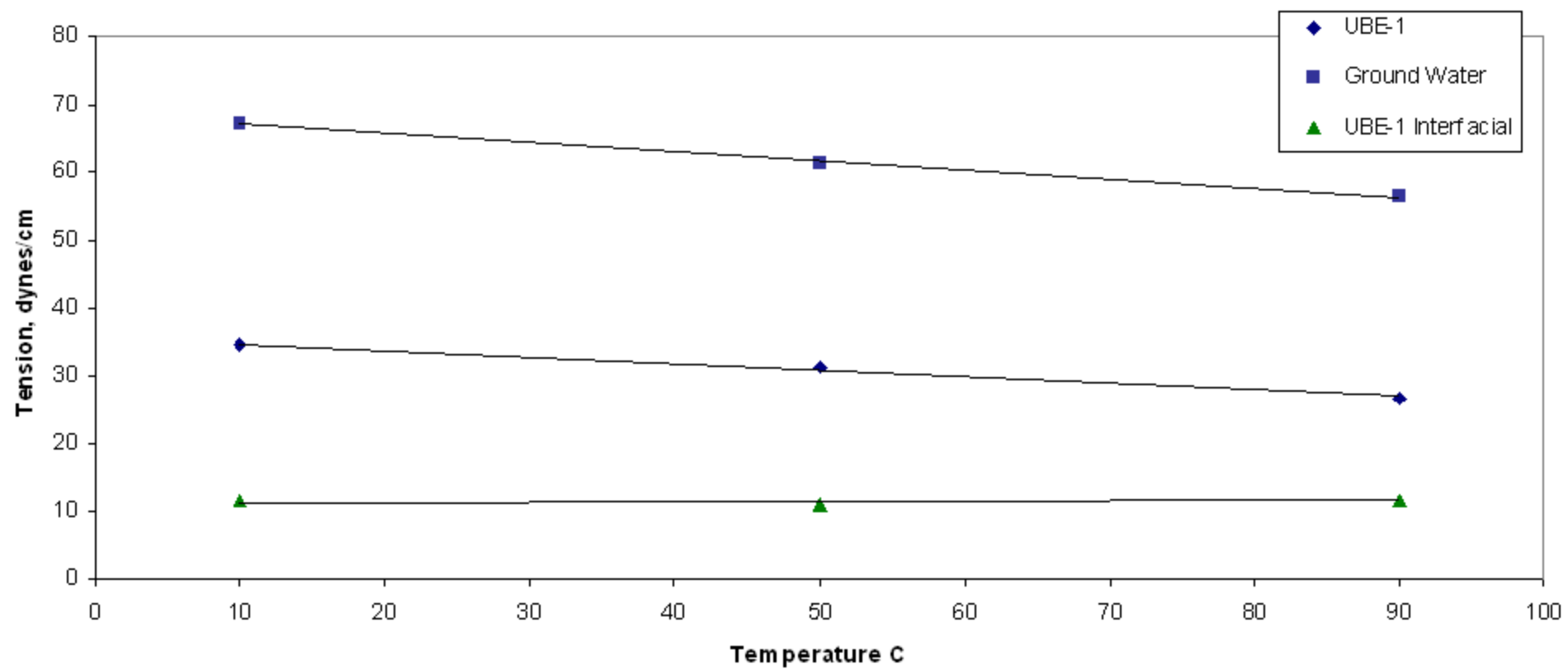


Figure 5. Photograph of Montrose DNAPL from well UBT-2 and RO water in the tensiometer setup.



**Figure 6. Temperature history during steam injection for Column #1.
SSB-6 88ft**

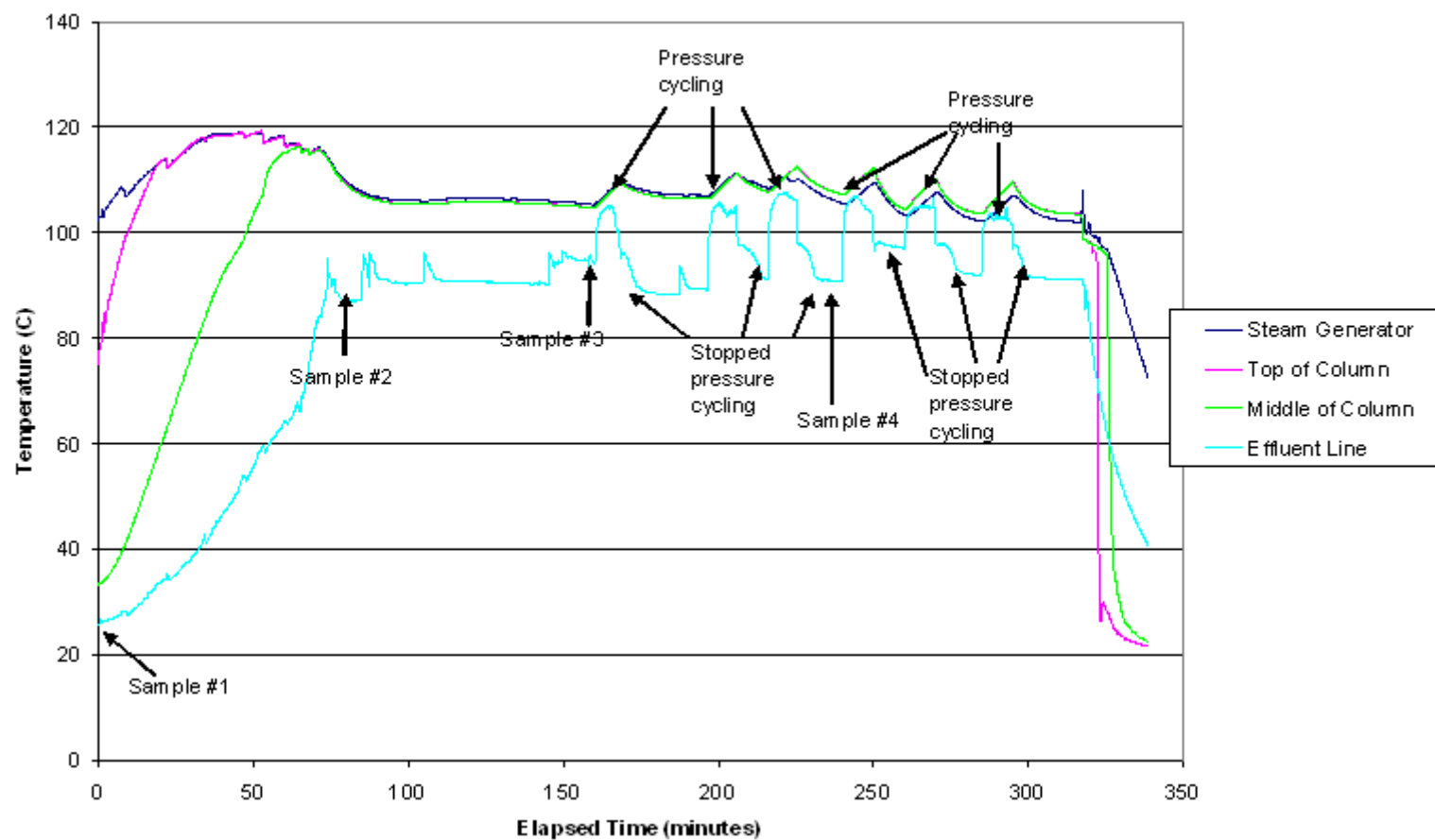


Figure 7. Initial and final soil concentrations for steam injection Column #1.
SSB-6 88ft

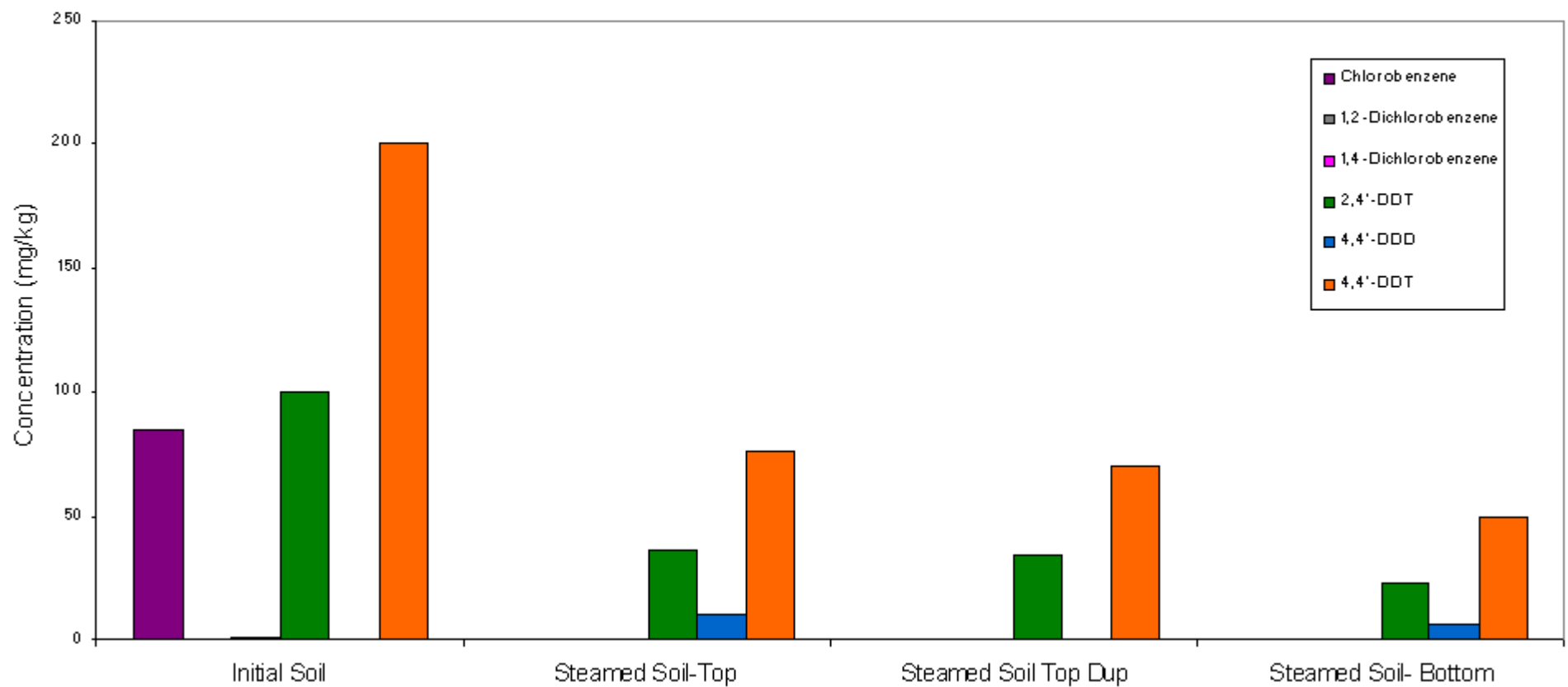
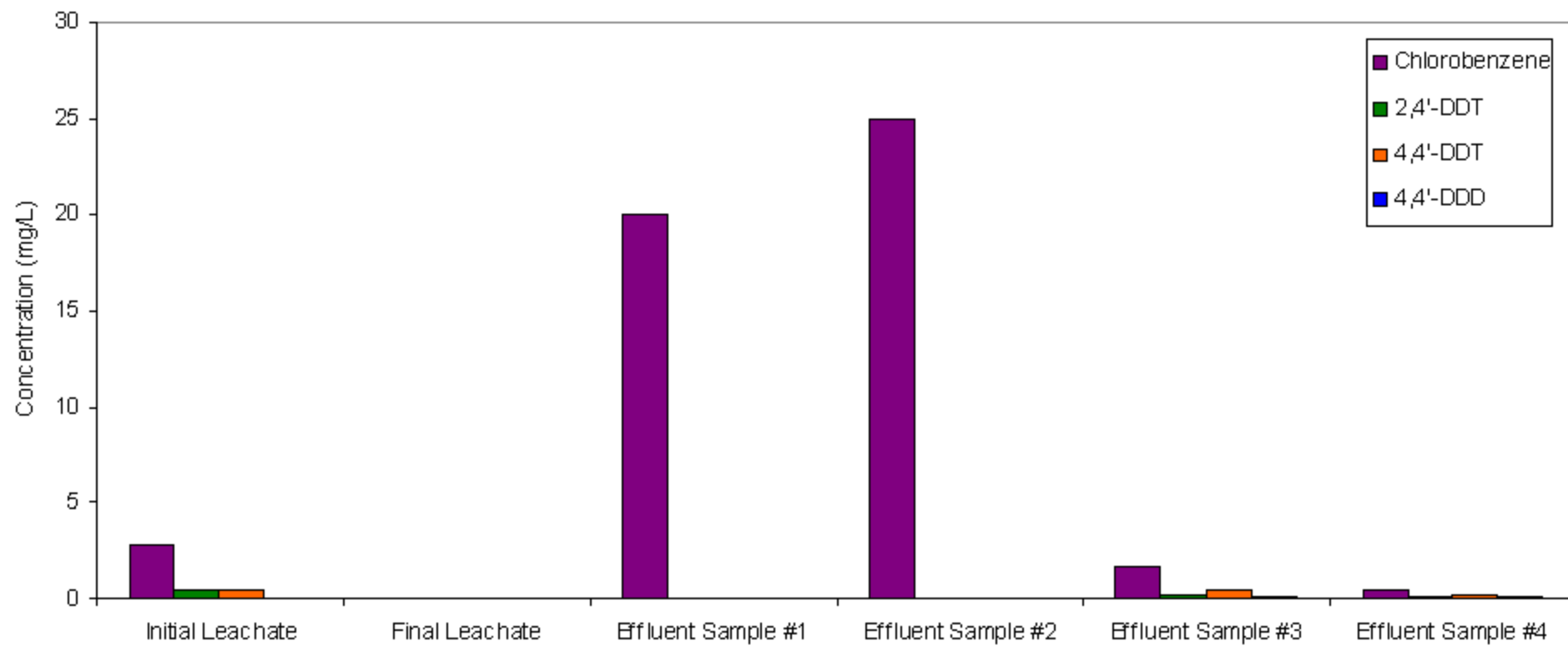


Figure 8. Aqueous concentrations for steam injection Column #1.
SSB-6 88ft



**Figure 9. Temperature history during steam injection for Column #2.
SSB-6 77 ft**

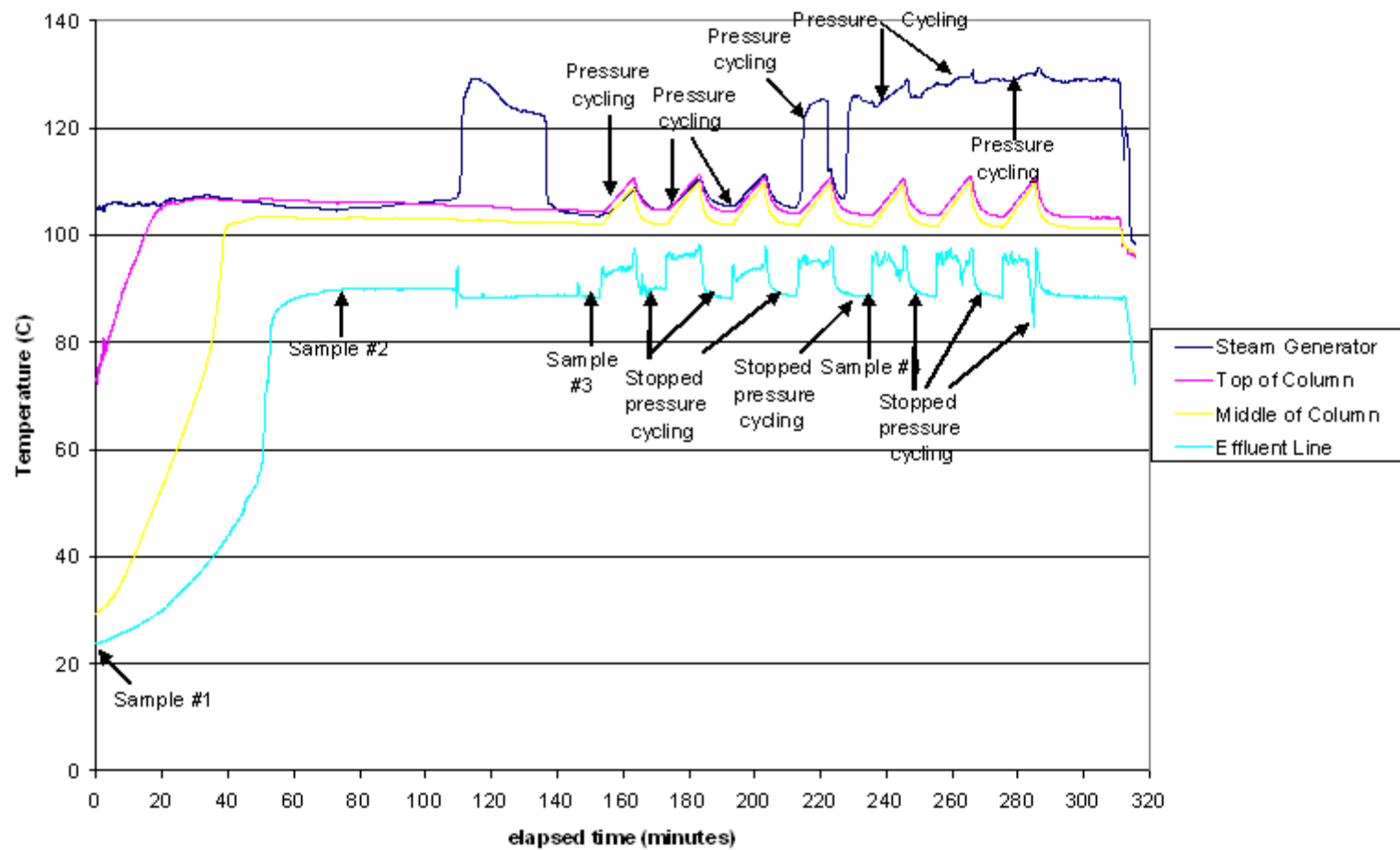


Figure 10. Initial and final soil concentrations for steam injection Column #2.
SSB-6 77ft

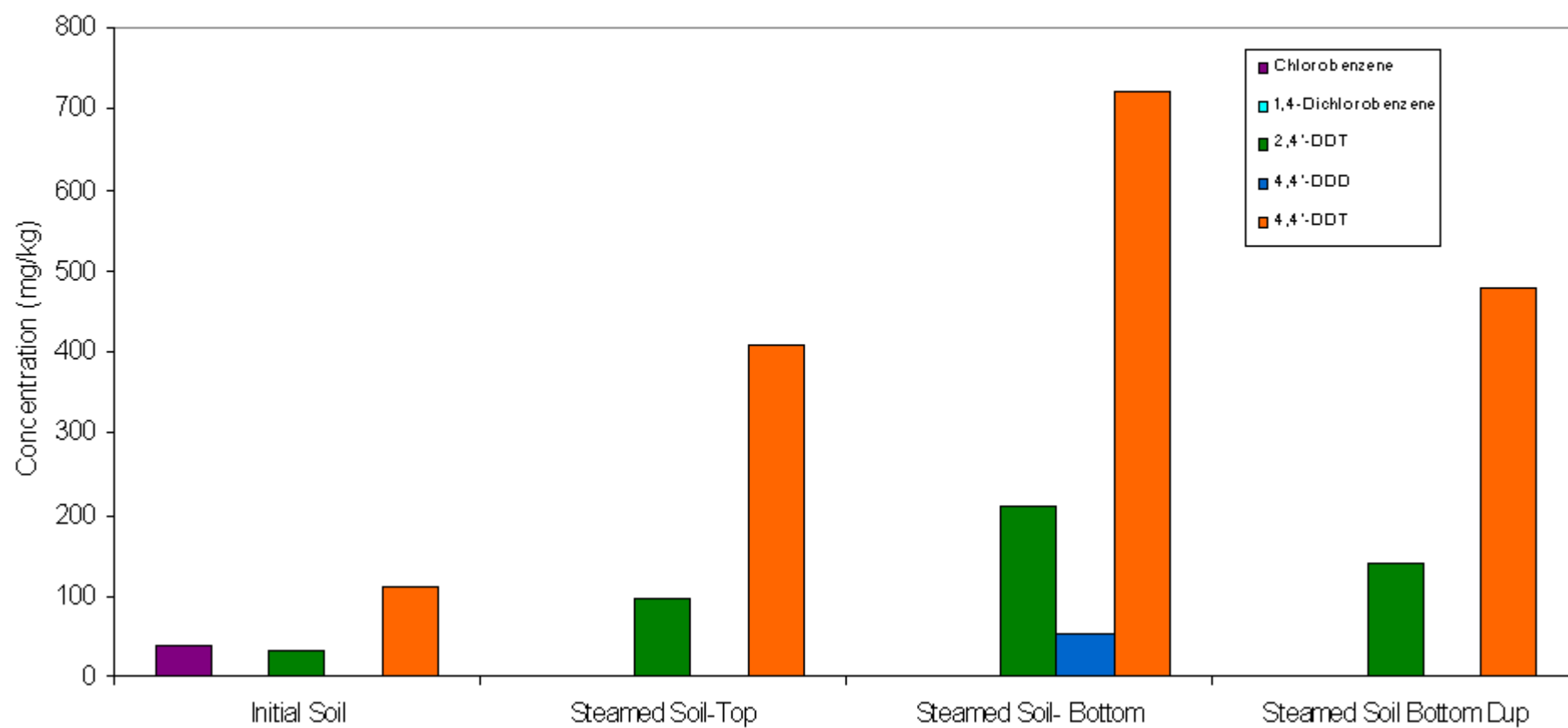


Figure 11. Aqueous concentrations for steam injection Column #2.
SSB-6 77ft

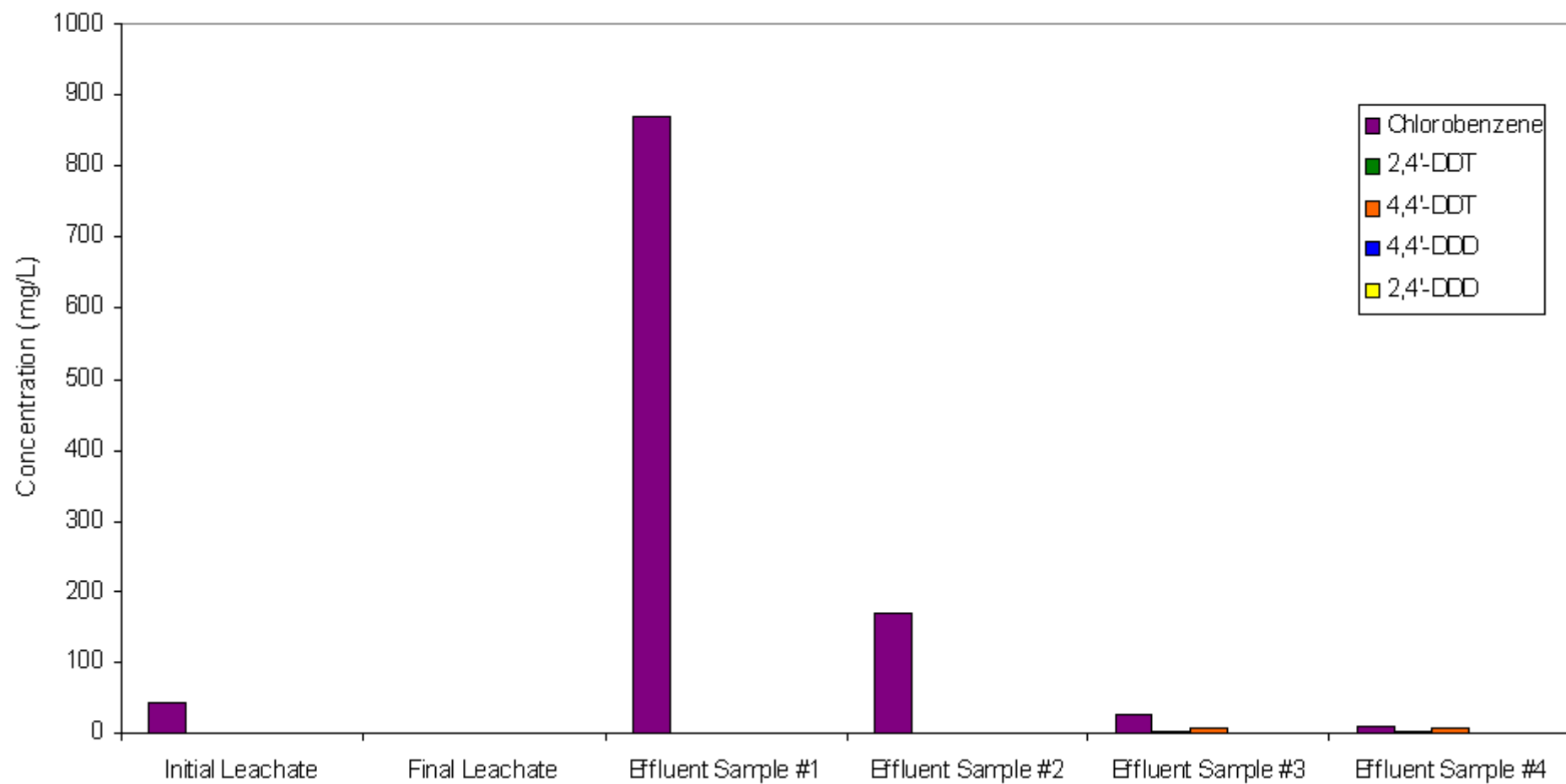


Figure 12. Temperature history during steam injection for Column #3
PSB-4 88ft

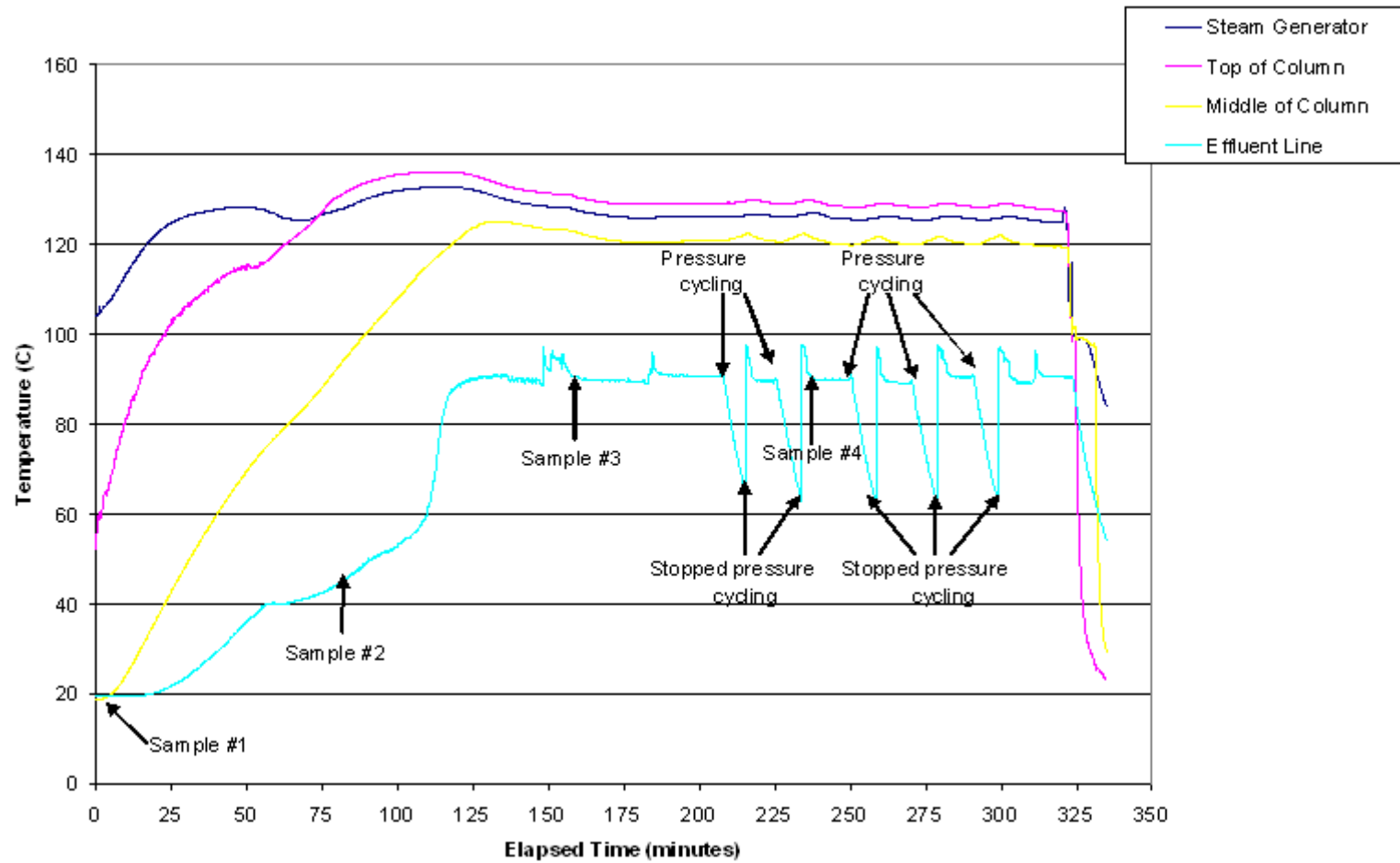


Figure 13. Initial and final soil concentrations for steaminjection Column #3
PBS-488ft

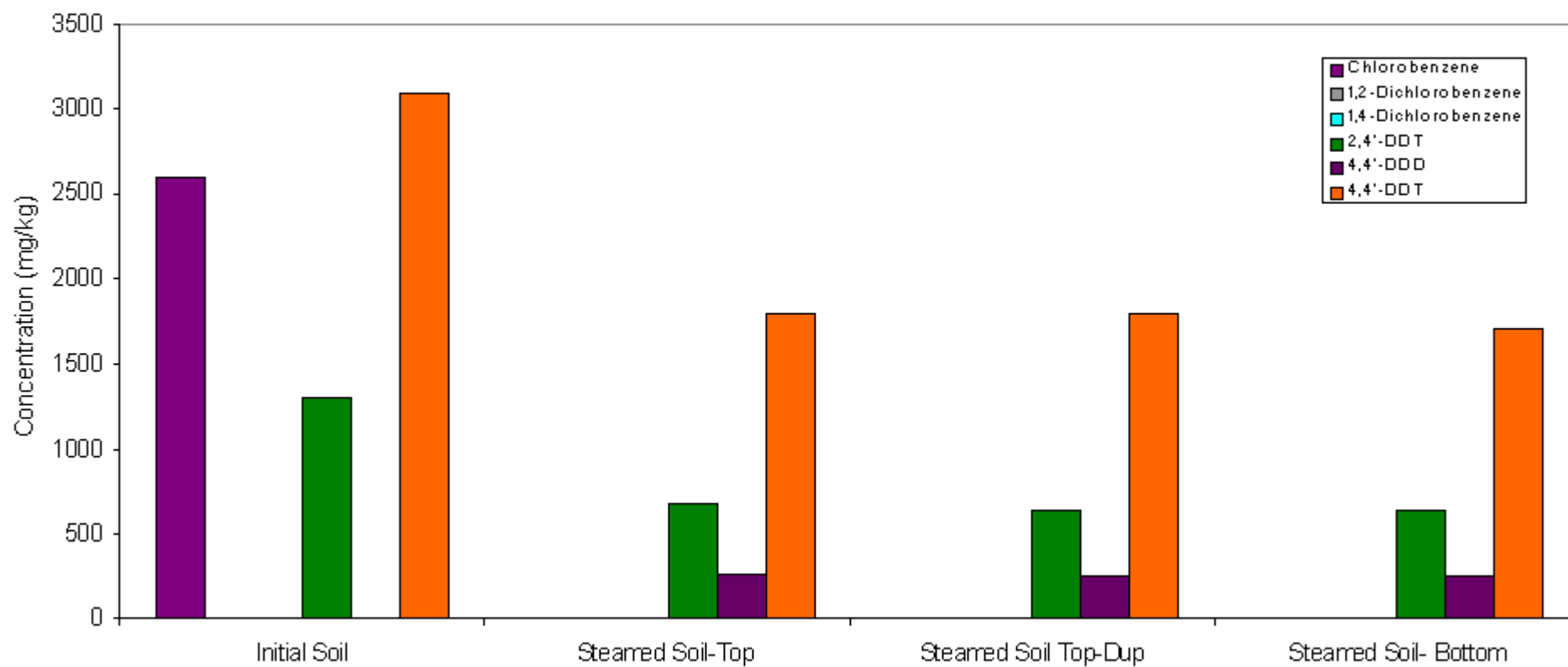


Figure 14. Aqueous concentration for steam injection Column #3.
FBS-4-88ft

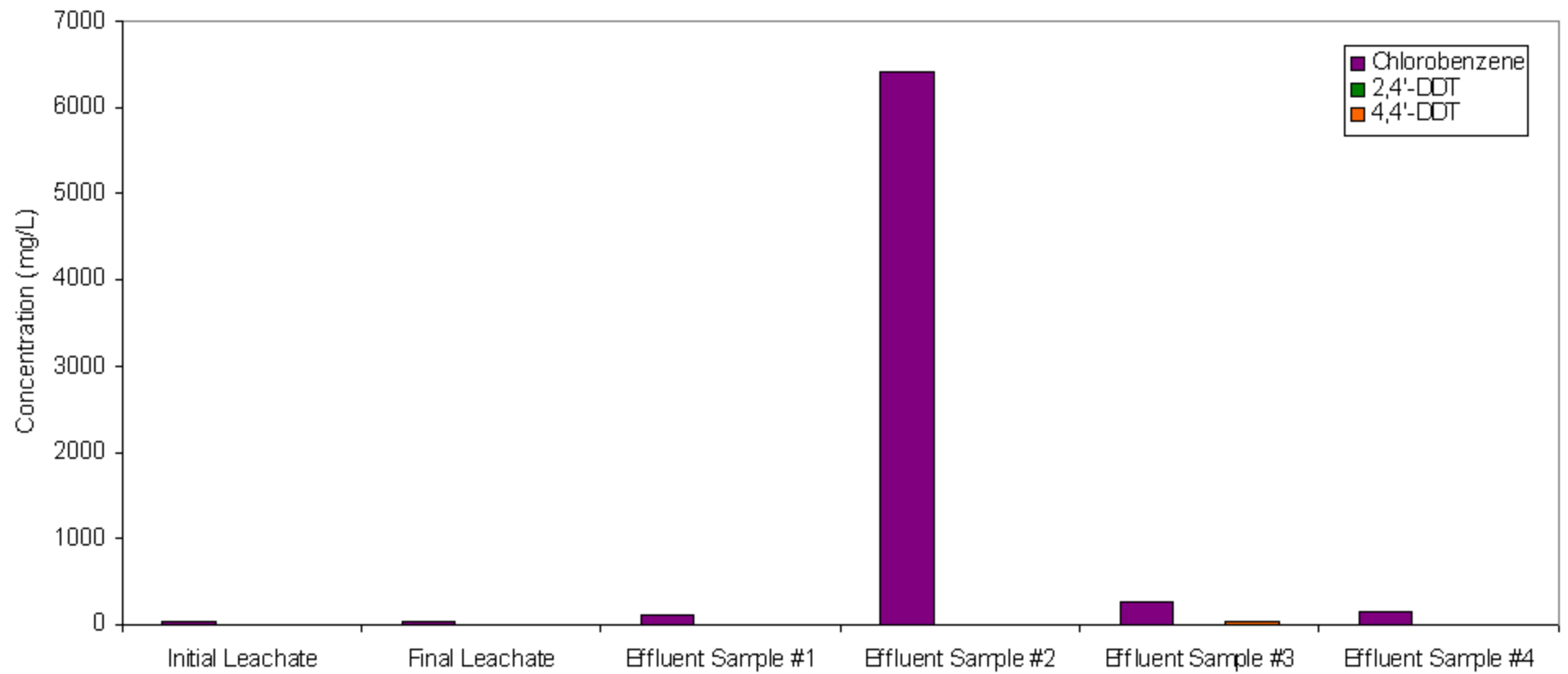


Figure 15. Temperature history during steam injection for Column #3 duplicate.
PSB-4 88ft

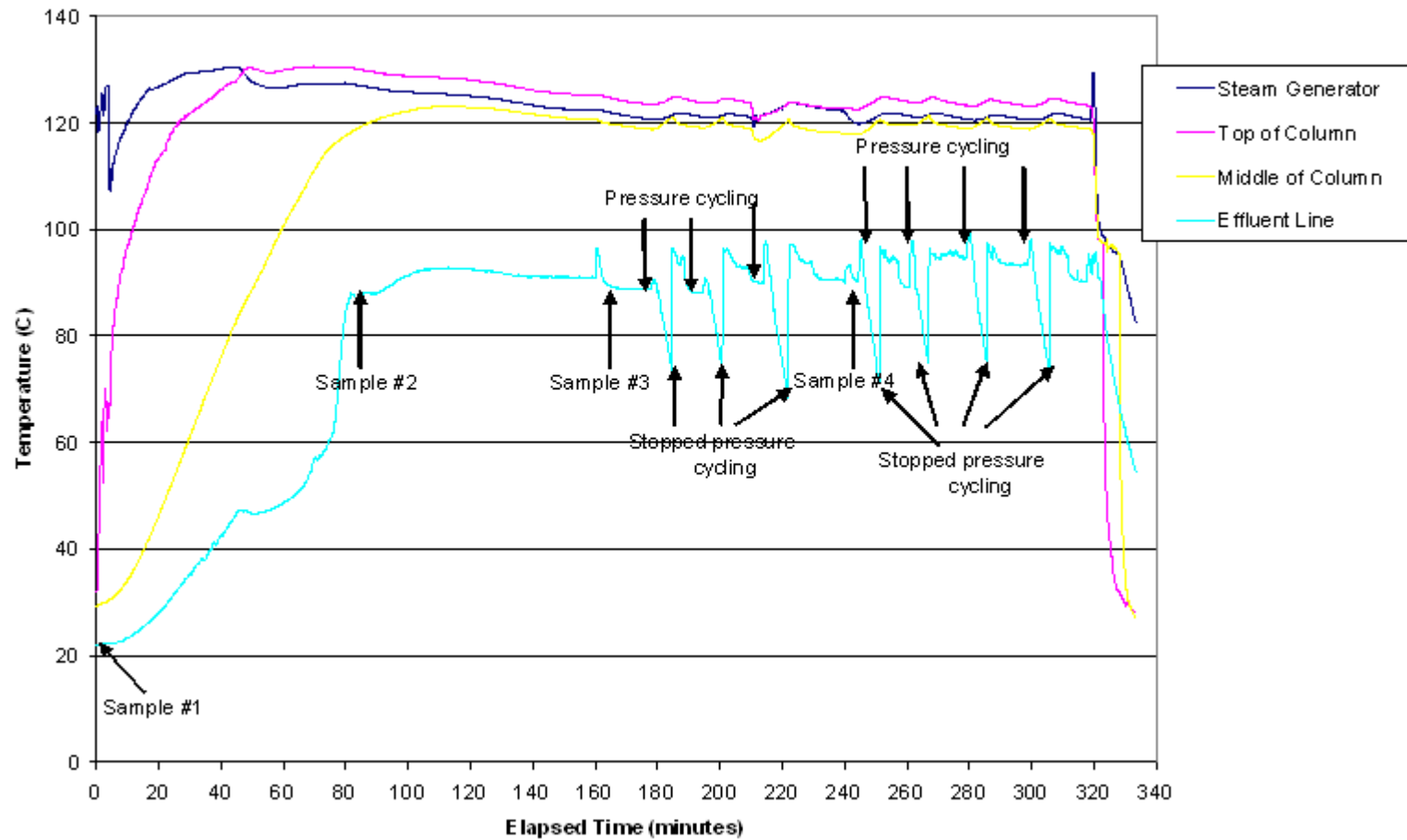


Figure 16. Initial and final soil concentrations for steam injection Column #3 duplicate.
PSB-4-88ft

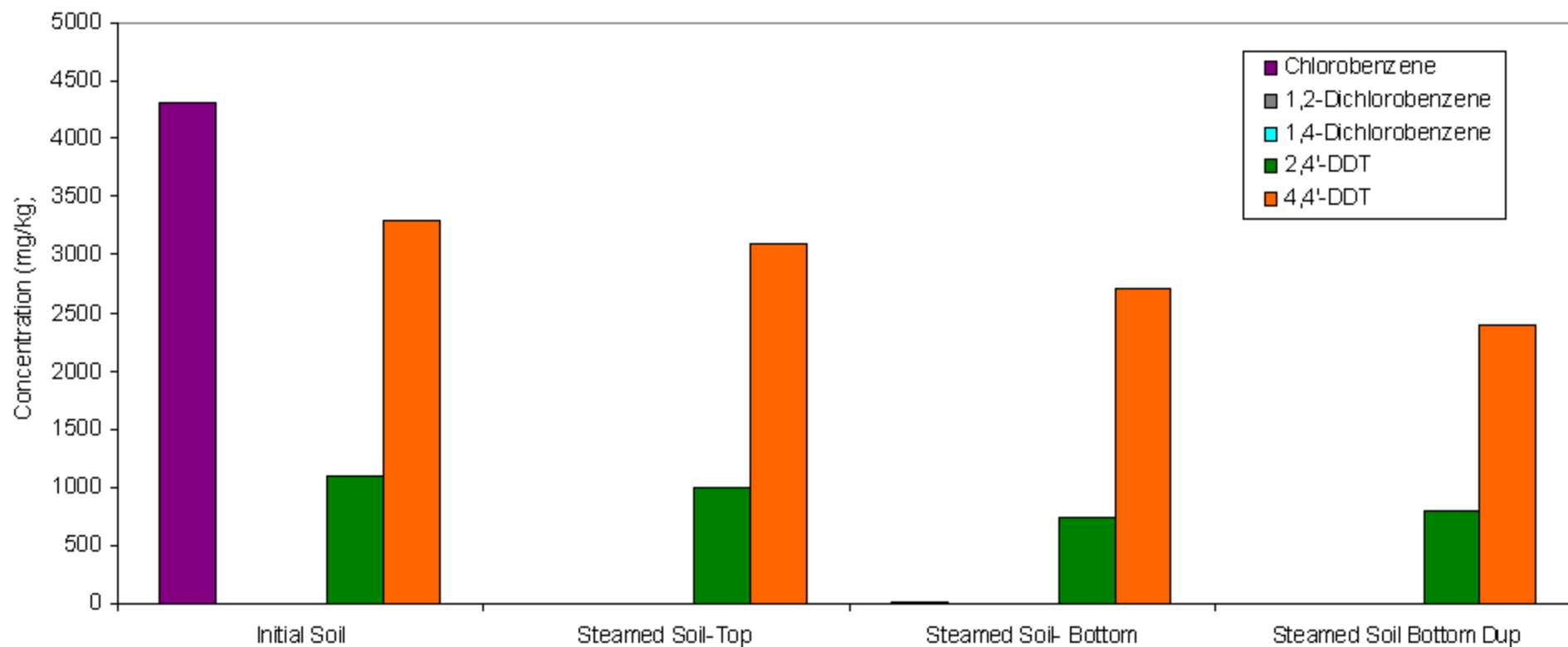
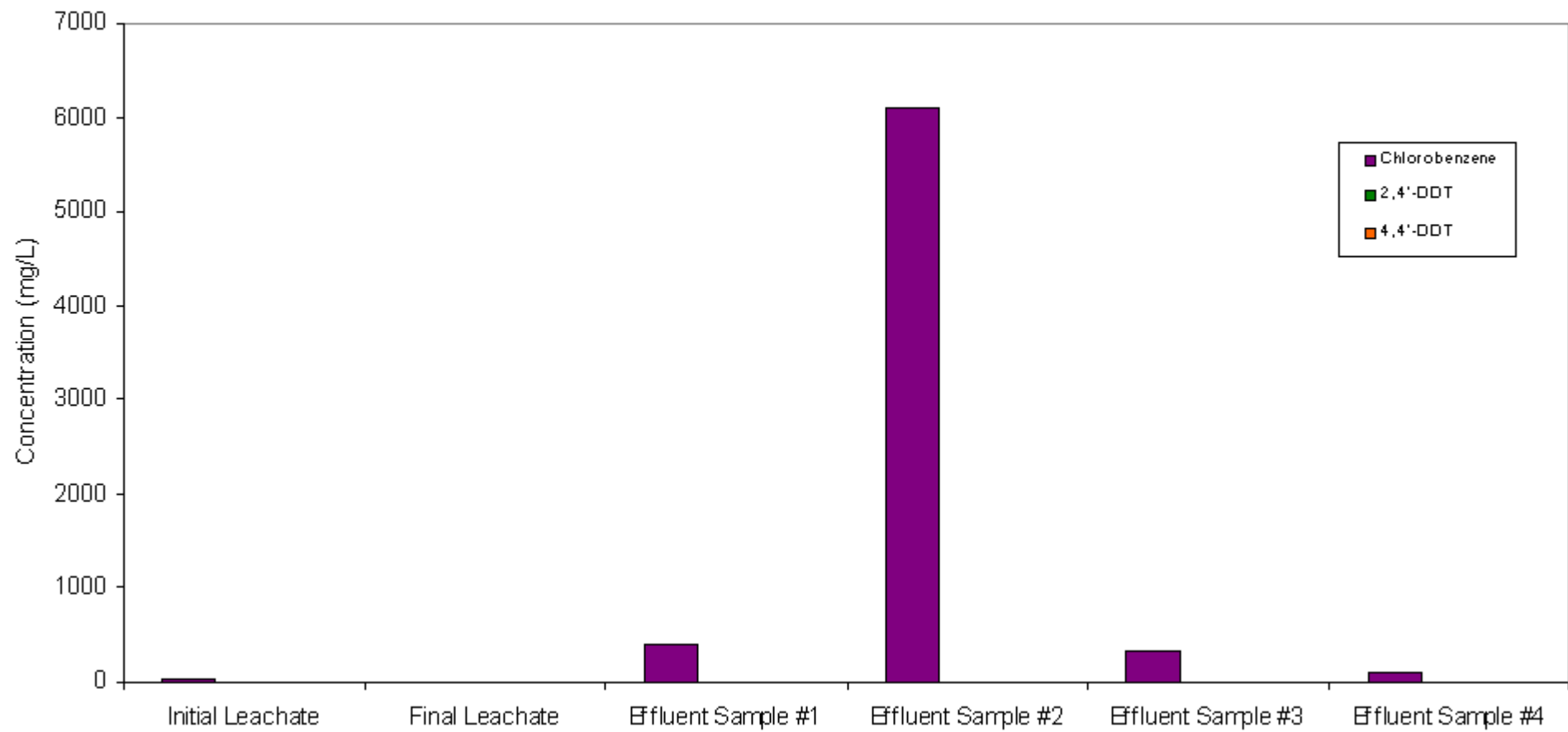


Figure 17. Aqueous concentrations for steam injection Column #3 duplicate.
PSB-4 88ft



**Figure 18. Temperature history during steam injection for Column #4.
PSB-15 75ft**

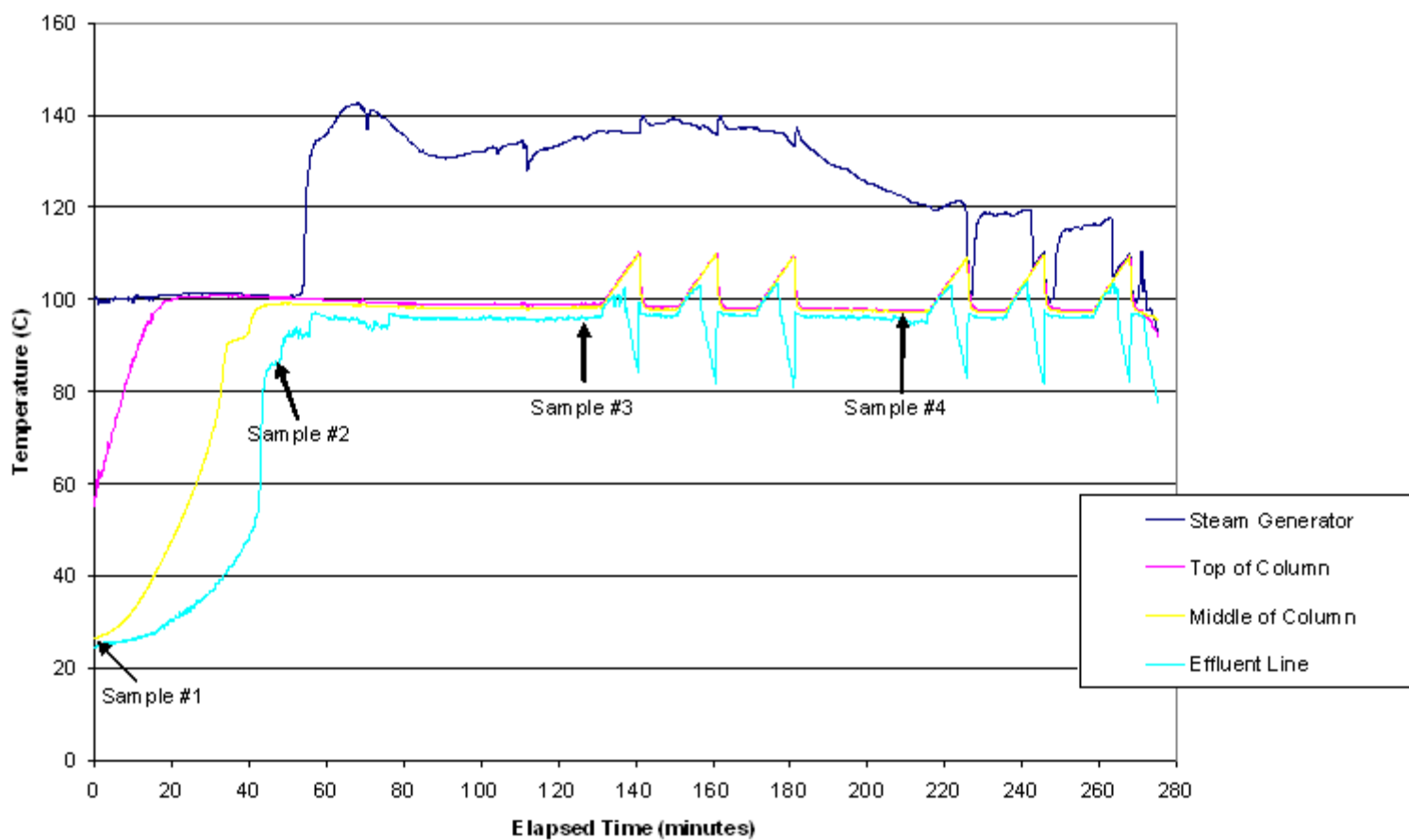


Figure 19. Initial and final soil concentrations for steam injection Column #4.
FSB- 15-75ft

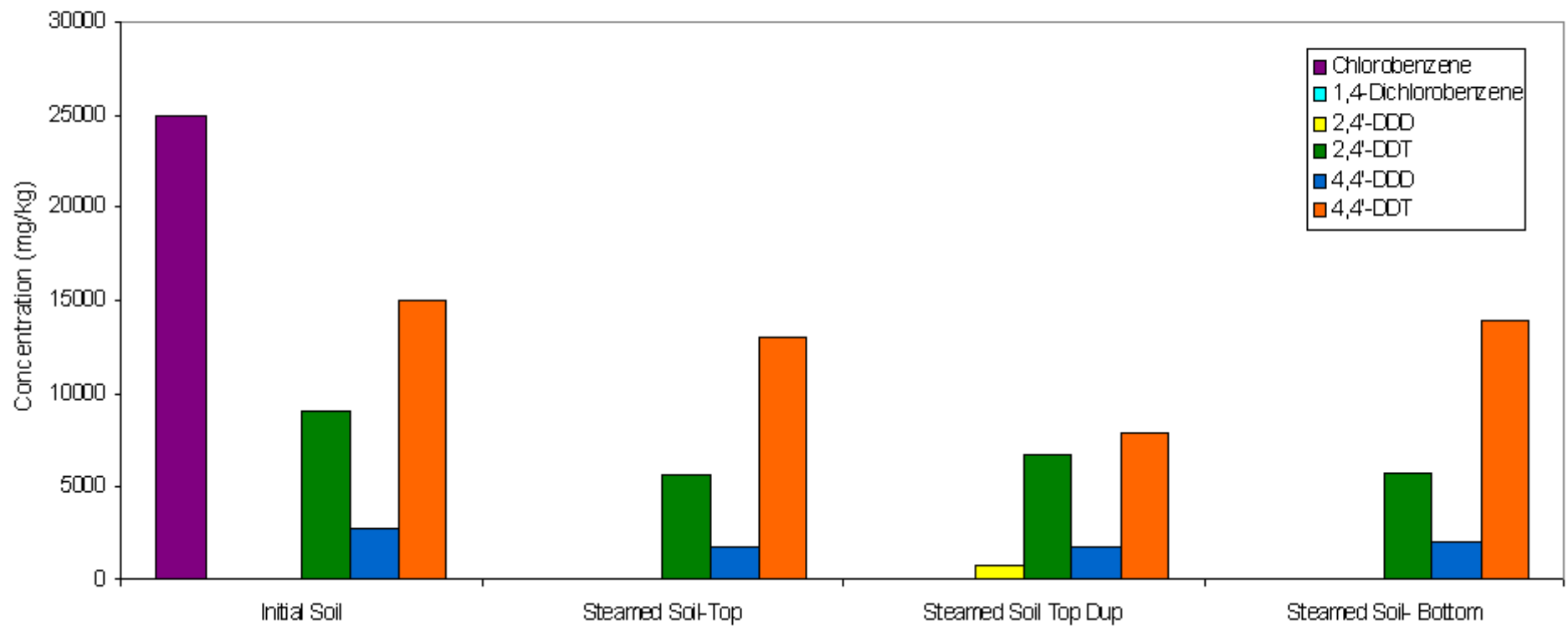
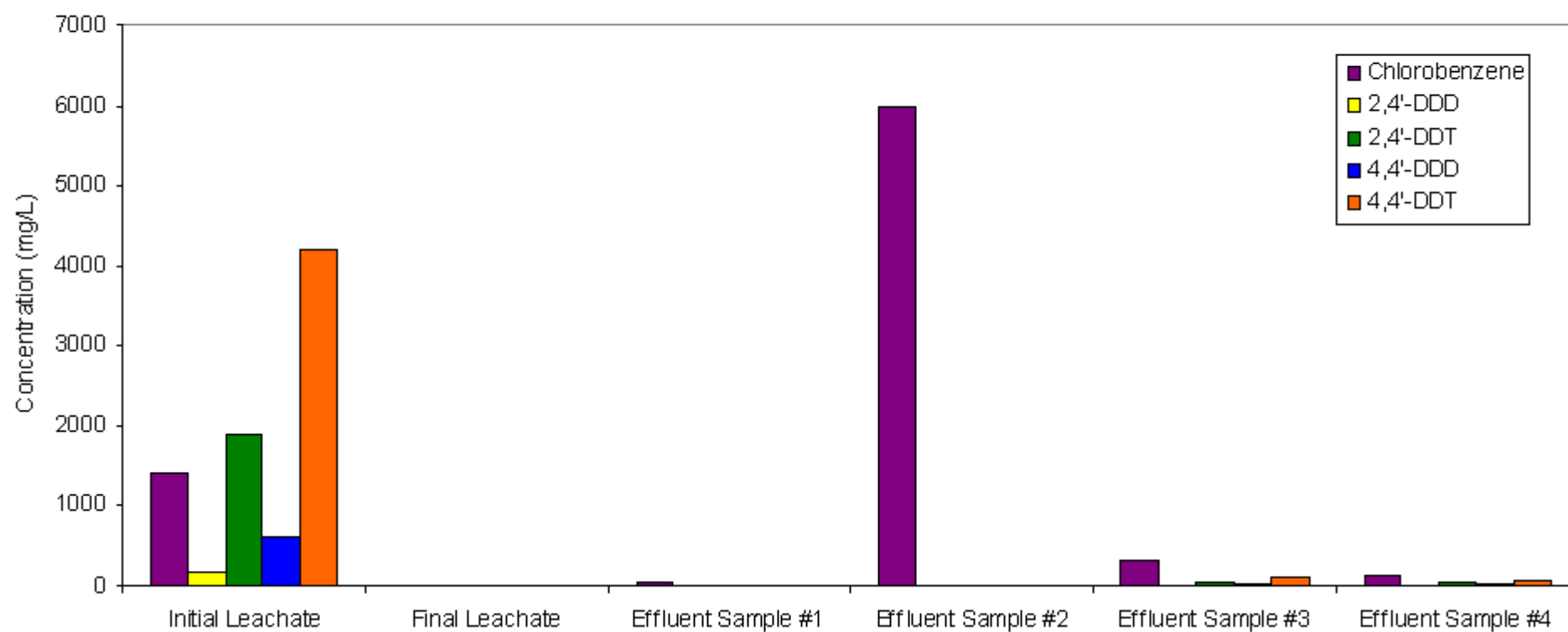


Figure 20. Aqueous concentrations for steam injection Column #4.
PSB-15-75ft



**Figure 21. Temperature history during steam injection for Column #5.
SSB-2 86.5 ft**

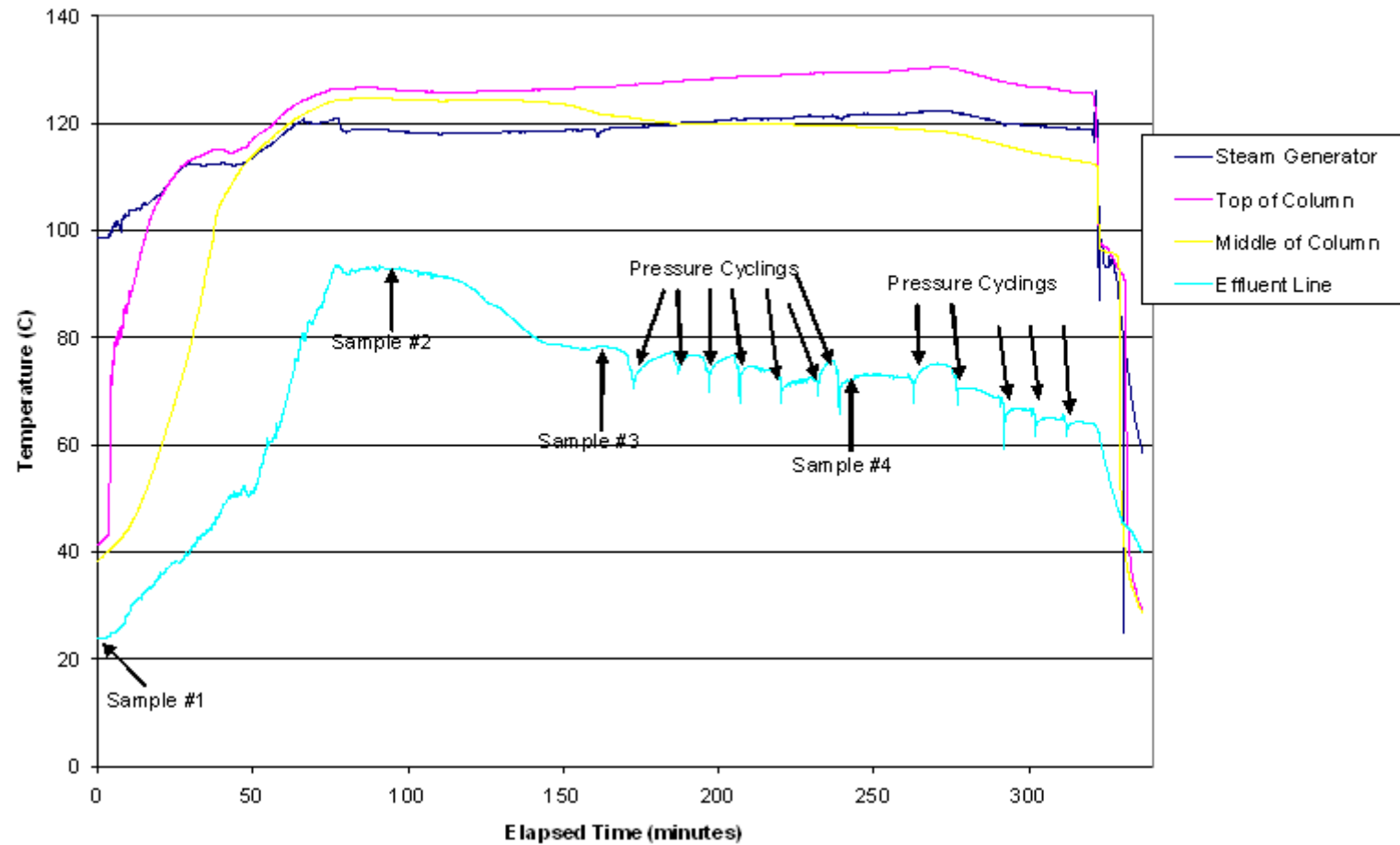


Figure 22. Initial and final soil concentrations for steam injection Column #5.
SSB-2 86.5ft

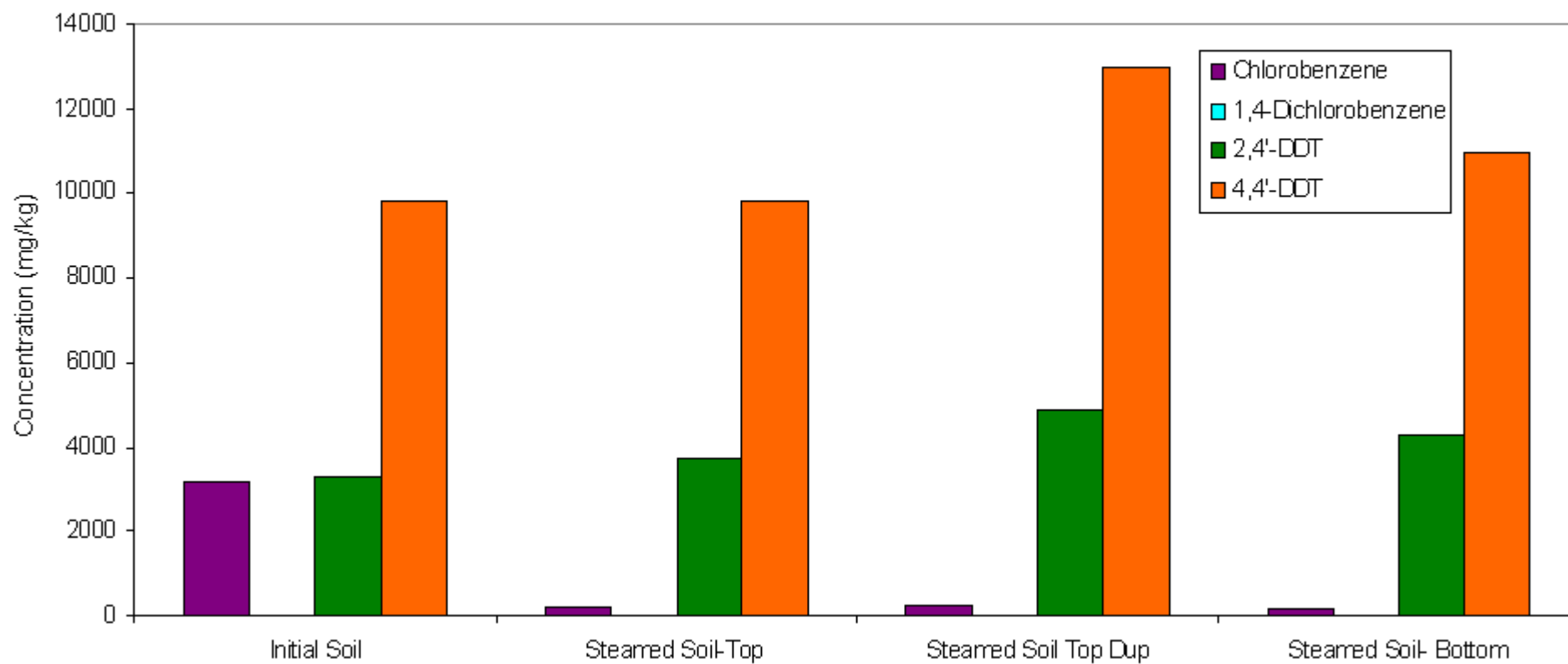


Figure 23. Aqueous concentrations for steam injection Column #5.
SSB-2 86.5ft

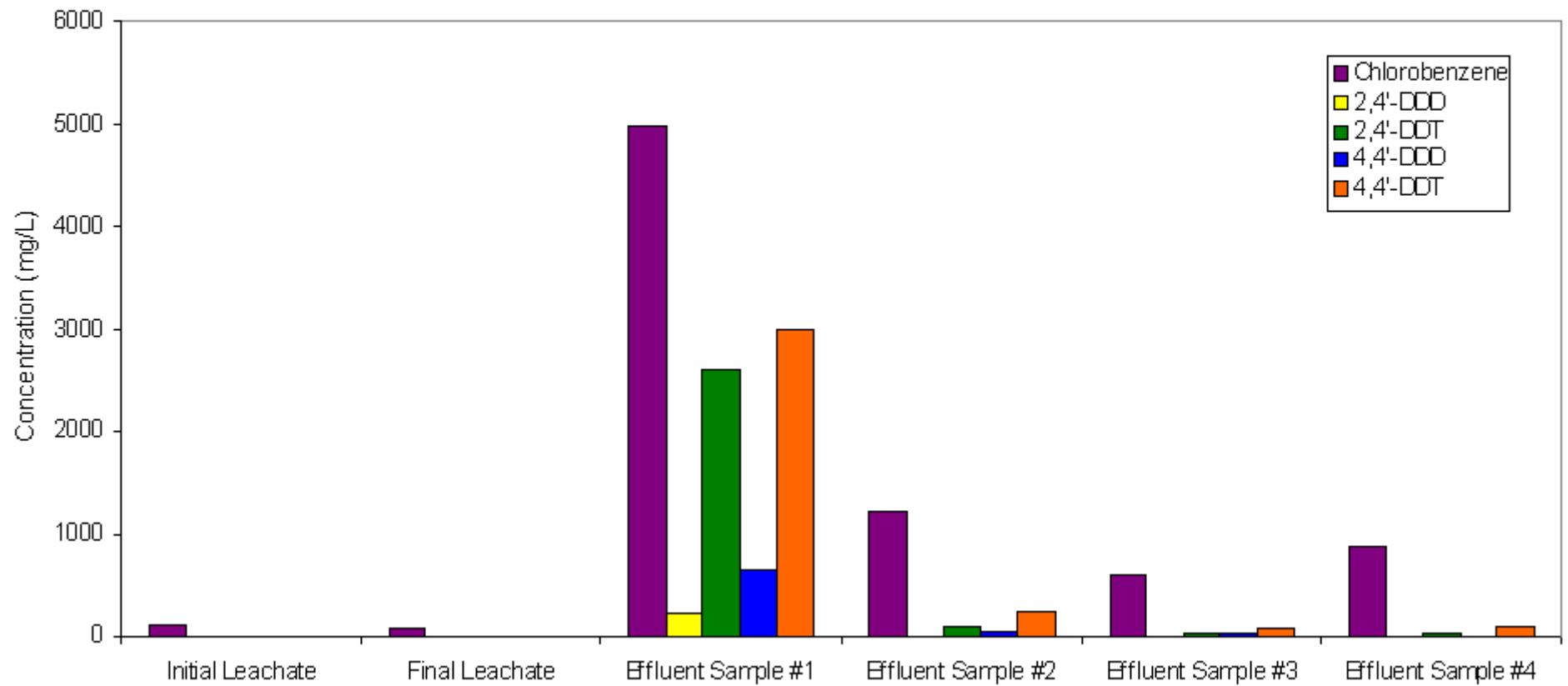


Figure 24. Comparison of initial and steamed soil chlorobenzene concentrations.

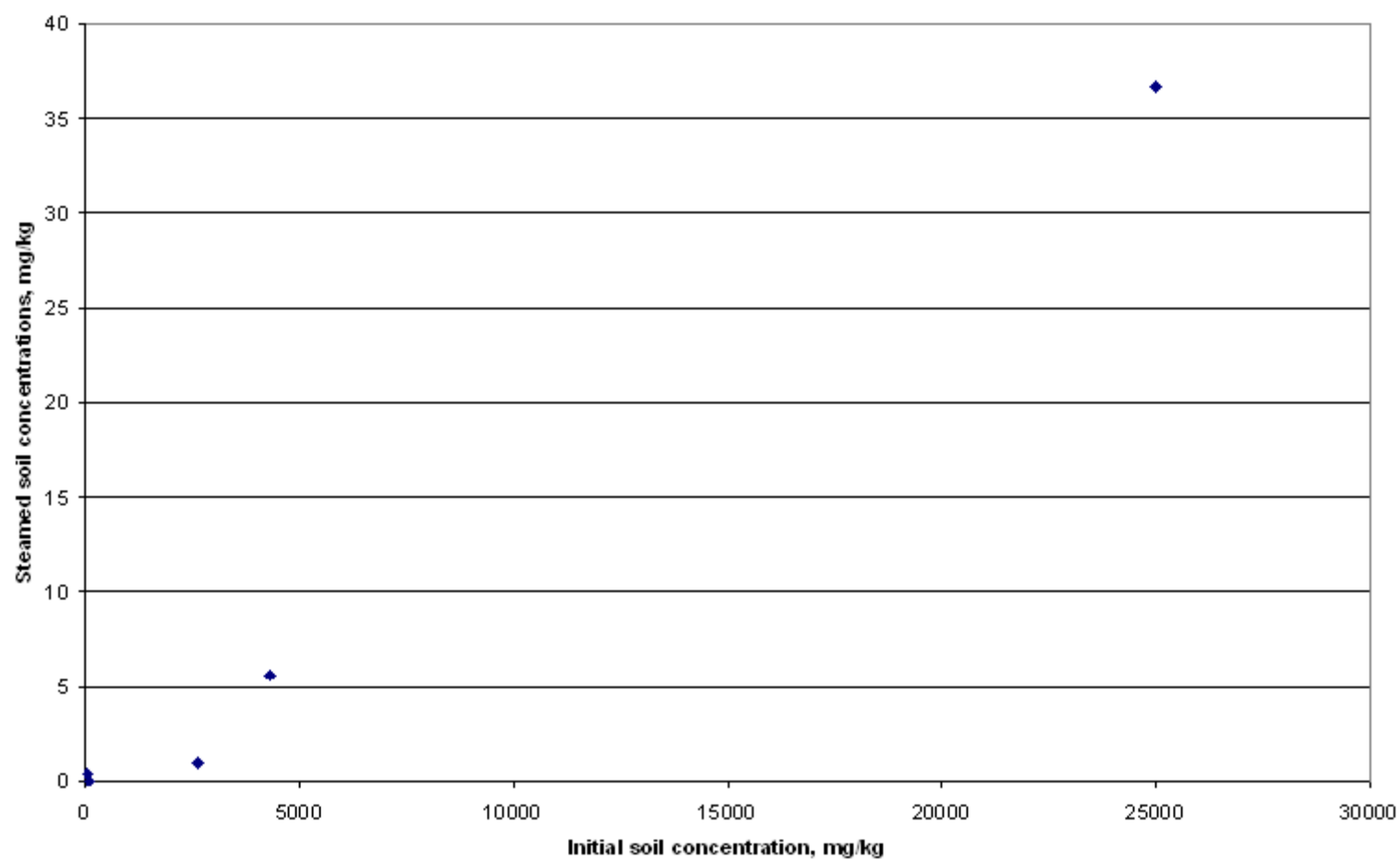


Figure 25. Weight of effluent collected as a function of the mass of steam injected for each of the column experiments

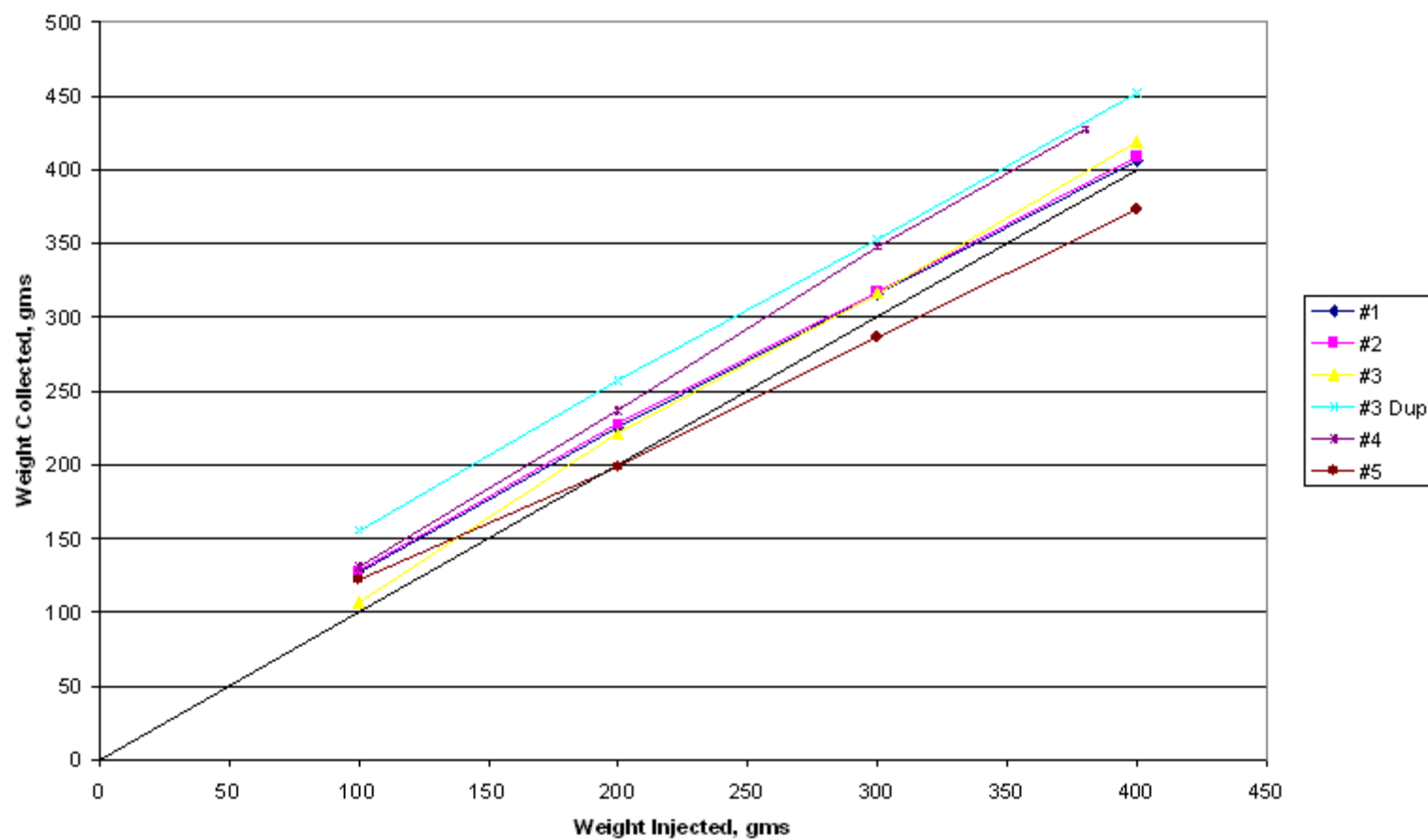
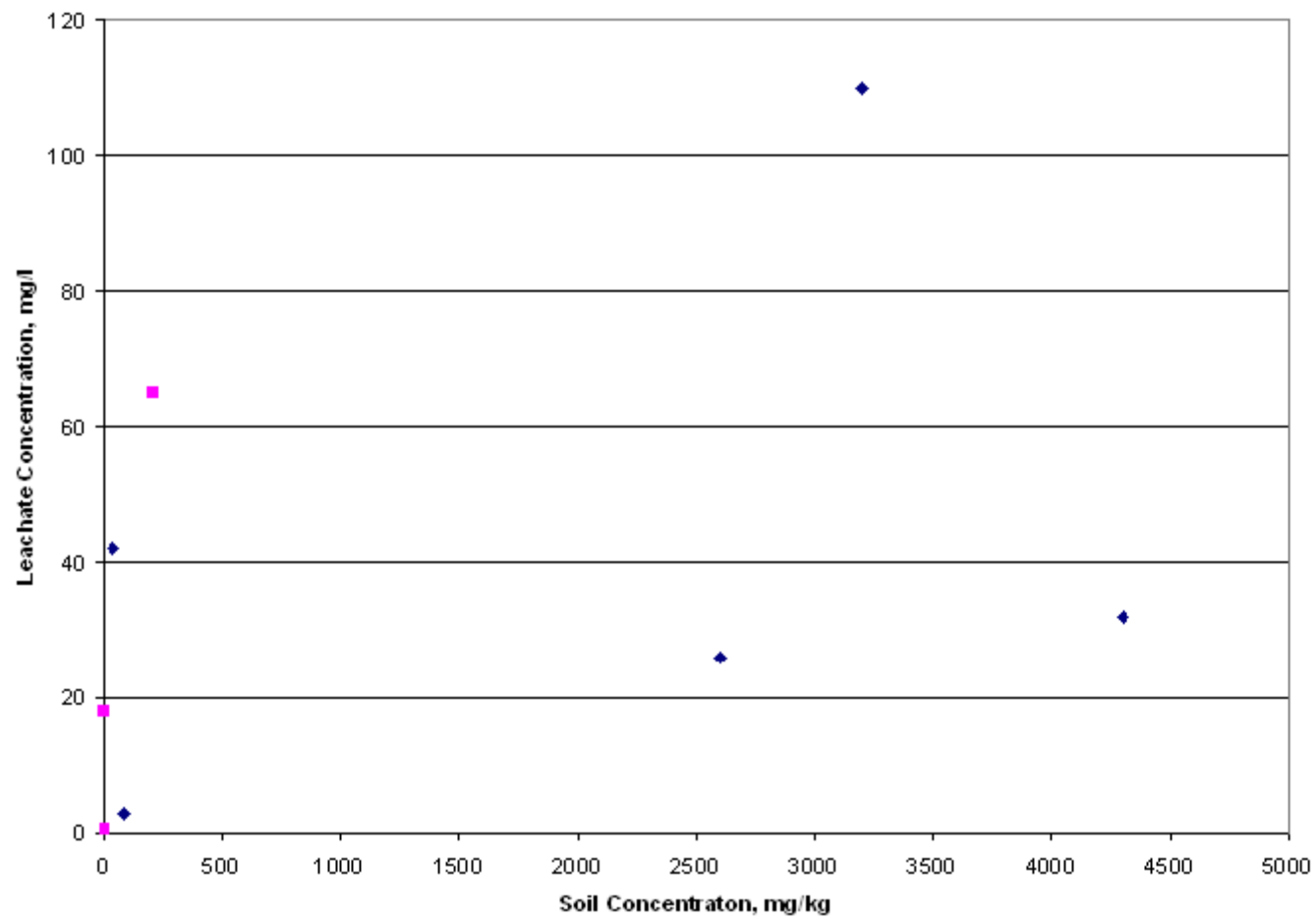


Figure 26. Leachate concentration as a function of soil concentration for initial and steamed soils.



APPENDIX A

Table A-1. Soil concentrations for Column #1 (mg/kg)

SSB-6 88 ft	Chlorobenzene	1,2-Dichlorobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Soil	85	<0.5	0.910	100	<50	200	300
Steamed Soil-Top	0.025	0.0014	0.011	36	10	76	122
Steamed Soil-Top-Duplicate	0.048	<0.00097	0.007	34	<10	70	104
Steamed Soil- Bottom	0.0061	<0.00096	<0.00096	23	6.7	50	79.7

Table A-2. Water concentrations for Column #1 (mg/l)

SSB-6 88 ft	Chlorobenzene	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Leachate	2.9	0.4	<0.1	0.45	0.85
Final Leachate	<0.1	<0.1	<0.1	<0.1	
Effluent Sample #1	20	<0.37	<0.37	<0.37	
Effluent Sample #2	25	<0.5	<0.5	<0.5	
Effluent Sample #3	1.6	0.26	0.11	0.42	0.79
Effluent Sample #4	0.49	0.16	0.074	0.260	0.494

Table A-3. Soil concentrations for Column #2 (mg/kg)

SSB-6 77 ft	Chlorobenzene	1,2-Dichloobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Soil	39	<0.5	0.6	32	<25	110	142
Steamed Soil-Top	0.11	<0.00095	0.0016	97	<38	410	507
Steamed Soil-Bottom	0.64	<0.1	<0.1	210	53	720	983
Steamed Soil- Bottom Duplicate	0.37	< 0.087	<0.087	140	<75	480	620

Table A-4. Water concentrations for Column #2 (mg/l)

SSB-6 77 ft	Chlorobenzene	2,4'-DDD	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Leachate	42	<1.2	<1.2	<1.2	<1.2	
Final Leachate	<0.12	<0.12	<0.12	<0.12	0.31	0.31
Effluent Sample #1	870	<19	<19	<19	<19	
Effluent Sample #2	170	<4.8	<4.8	<4.8	<4.8	
Effluent Sample #3	25	0.6	4.8	1.0	7.4	13.8
Effluent Sample #4	9	0.6	4.0	1.0	6.1	11.7

Table A-5. Soil concentrations for Column #3 (mg/kg)

PSB-4-88 ft	Chlorobenzene	1,2-Dichlorobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Soil	2,600	<20	<20	1,300	<750	3,100	4,400
Steamed Soil-Top	0.69	<0.1	0.16	680	260	1,800	2,740
Steamed Soil-Top-Duplicate	0.59	<0.1	0.24	640	250	1,800	2,690
Steamed Soil- Bottom	1.7	0.16	0.94	640	250	1,700	2,590

Table A-6. Water concentrations for Column #3 (mg/l)

PSB-4-88 ft	Chlorobenzene	2,4'-DDT	4,4'-DDT	Total DDT
Initial Leachate	26	<0.5	<0.5	
Final Leachate	18	<0.56	<0.56	
Effluent Sample #1	100	<2.4	<2.4	
Effluent Sample #2	6,400	<220	<220	
Effluent Sample #3	250	10	15	25
Effluent Sample #4	150	<4.8	<4.8	

Table A-7. Soil concentrations for Column #3 duplicate (mg/kg)

PSB-4-88 ft	Chlorobenzene	1,2-Dichloobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDT	Total DDT
Initial Soil	4,300	<40	<40	1,100	3,300	4,400
Steamed Soil-Top	1.6	<0.1	0.3	1,000	3,100	4,100
Steamed Soil-Bottom	9.3	0.25	1.3	750	2,700	3,450
Steamed Soil- Bottom Duplicate	6.0	<0.2	0.89	800	2,400	3,200

Table A-8. Water concentrations for Column #3 duplicate (mg/l)

PSB-6 88 ft	Chlorobenzene	2,4'-DDT	4,4'-DDT	Total DDT
Initial Leachate	32	<1.2	<1.2	
Final Leachate	0.5	<0.096	0.13	0.13
Effluent Sample #1	410	<6.4	6.9	6.9
Effluent Sample #2	6,100	<100	<100	
Effluent Sample #3	320	<5.3	6.2	6.2
Effluent Sample #4	97	3.1	7.2	10.3

Table A-9. Soil concentrations for Column #4 (mg/kg)

PSB-15-75 ft	Chlorobenzene	1,4-Dichlorobenzene	2,4'-DDD	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Soil	25,000	<670	<1400	9,000	2,800	15,000	26,800
Steamed Soil-Top	35	0.95	<3000	5,600	1,700	13,000	20,300
Steamed Soil- Top Duplicate	37	0.77	830	6,800	1,700	7,900	17,230
Steamed Soil- Bottom	38	0.87	<3000	5,700	2,100	14,000	21,800

Table A-10. Water concentrations for Column #4 (mg/l)

PSB-15-75 ft	Chlorobenzene	2,4'-DDD	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Leachate	1,400	190	1,900	610	4,200	6,900
Final Leachate	<0.071	<0.071	<0.071	<0.071	<0.071	
Effluent Sample #1	53	<1	<1	<1	<1	
Effluent Sample #2	6,000	<230	<230	<230	<230	
Effluent Sample #3	320	<9.1	66	17	100	183
Effluent Sample #4	140	8.2	58	14	68	148.2

Table A-11. Soil concentrations for Column #5 (mg/kg)

SSB-2 86.5 ft	Chlorobenzene	1,4-Dichlorobenzene	2,4'-DDT	4,4'-DDT	Total DDT
Initial Soil	3,200	<100	3,300	9,800	13,100
Steamed Soil-Top	200	2	3,700	9,800	13,500
Steamed Soil- Top Duplicate	260	3	4,900	13,000	17,900
Steamed Soil- Bottom	170	<2	4,300	11,000	15,300

Table A-12. Water concentrations for Column #5 (mg/l)

SSB-2 86.5 ft	Chlorobenzene	2,4'-DDD	2,4'-DDT	4,4'-DDD	4,4'-DDT	Total DDT
Initial Leachate	110	<2.6	<2.6	<2.6	<2.6	
Final Leachate	65	<1.2	2.7	1.6	6.9	11.2
Effluent Sample #1	5,000	230	2,600	640	3,000	6,470
Effluent Sample #2	1,200	<26	87	37	250	374
Effluent Sample #3	600	<11	26	15	76	117
Effluent Sample #4	880	<23	30	<23	94	124

APPENDIX B1

Standard Operating Procedure

Density Measures as a Function of Temperature

Disclaimer:

This standard operating procedure has been prepared for the use of the Ground Water and Ecosystems Restoration Division of the U.S. Environmental Protection Agency and may not be specifically applicable to the activities of other organizations. **THIS IS NOT AN OFFICIAL EPA APPROVED METHOD.** This document has not been through the Agency's peer review process or ORD clearance process.

1.0 Purpose (Scope and Application)

To discover the density of a fluid sample using a 25 ml volumetric flask. The measurements are made at a constant temperature ranging from 10 degrees centigrade to 90 degrees centigrade.

2.0 Method Summary

A measurement in grams/cm³ is done on a 25 ml volumetric flask. The liquid sample to be measured is placed inside the flask, to a volume that is below the volumetric line of the flask. This container is placed inside a water bath and allowed to reach a constant temperature. The volume in the flask is then adjusted to the volumetric line. The container is weighed and a density measurement is calculated using the formula $D=M/V$, where M is mass and V is volume. This procedure is repeated with two other samples and the final density comes from the average of the three measurements.

3.0 Reagents

N/A

4.0 Equipment and Apparatus

Water bath capable of holding constant temperatures between 10 and 90 degrees centigrade. Three 25 ml volumetric flasks. Laboratory balance capable of weighing to the nearest 0.001g. NIST-traceable thermometer to verify water bath temperature.

5.0 Health and Safety Precautions

- 5.1 When using the water bath, caution needs to be taken to prevent burns from high temperatures.
- 5.2 When testing volatile liquids, the water bath needs to be setup inside the hood to prevent exposure.

6.0 Interferences

- 6.1 Once a sample has been placed inside the water bath, 1 hour should be allowed for equilibration of the sample to temperature. If the sample is not equilibrated to temperature the density will be inaccurate.
- 6.2 When reading the meniscus inside the flask at high temperatures the heat capacity of the sample needs to be considered. Holding samples with low heat capacities outside the water bath will influence the volume measurement.
- 6.3 The opening of the volumetric flask needs to be securely covered to ensure that the volatile components do not escape.

7.0 Procedure

- 7.1 Setup for the density measurement.
 - 7.1.1 Weigh (to the nearest 0.001 gms) and record the weight of the flask inside the lab book.
 - 7.1.2 Add the fluid sample to the flask, so that the volume is below the 25 ml line on the flask.
 - 7.1.3 Using a ring stand, suspend the flask inside the water bath at a constant temperature for one hour. Suspend a small beaker containing the same sample inside the water bath.
 - 7.1.4 Begin at the highest temperature at which measurements are to be made because the liquid will occupy the largest volume at the higher temperatures. After completing the measurement at the highest temperature, decrease the temperature to the next desired temperature measurement, and additional sample will need to be added to reach the 25 ml mark.

7.2 Making a Density Measurement

- 7.2.1 Once the sample has reached equilibrium with the temperature of the water bath, observe the volume measurement on the flask.
- 7.2.2 Using a micropipette, add the appropriate amount of sample from the reserve beaker to the flask so that the meniscus is on the 25 ml mark.
- 7.2.3 Dry the flask of all moisture from the water bath using a toilette.
- 7.2.4 Weigh the flask containing the fluid sample.
- 7.2.5 Subtract the empty weight of the flask from the combined weight of the flask and sample to arrive at the sample weight.
- 7.2.6 Using the formula $D=M/V$, divide the sample weight by the volume (25 ml).

8.0 QA/QC

- 8.1 Samples are measured three times and the average is taken and the standard deviation is calculated.

9.0 Calculations

Division is used to divide the mass of the sample by the volume that the sample occupies. An average is also taken between the three repeated measurements for the final density. The density is given in grams/centimeter cubed.

10.0 Miscellaneous Notes

N/A

11.0 References

ASTM D1217-93

APPENDIX B2

Standard Operating Procedure

Fisher Surface Tensiometer Model 20 Use and Maintenance

Disclaimer:

This standard operating procedure has been prepared for the use of the Ground Water and Ecosystems Restoration Division of the U.S. Environmental Protection Agency and may not be specifically applicable to the activities of other organizations. **THIS IS NOT AN OFFICIAL EPA APPROVED METHOD.** This document has not been through the Agency's peer review process or ORD clearance process.

1.0 Purpose (Scope and Application)

This SOP covers procedures for the calibration checks and for measuring surface and interfacial tension using the Fisher Surface Tensiometer Model 20.

2.0 Method Summary

After calibration is completed, a sample liquid is placed on the sample table until the platinum-iridium ring is submersed in the sample liquid. By manipulating the dials on the tensiometer a surface tension measurement or interfacial tension measurement will be recorded in Dynes per centimeter.

3.0 Reagents

N/A

4.0 Equipment and Apparatus

4.1 Fisher Surface Tensiometer Model 20

4.2 Weights of a known mass are needed. A 600 milligram weight is recommended to simplify calculations.

4.3 Sample containers with a diameter of no less than 45 millimeters. Containers should be cleansed with an acid wash.

5.0 Health and Safety Precautions

- 5.1 When using the water bath, caution needs to be taken to prevent burns from high temperatures.
- 5.2 When measuring hazardous fluids, the viscometer and water bath need to be setup inside the hood to prevent exposure.

6.0 Interferences

- 6.1 Samples tested in containers with a diameter smaller than 45 millimeters may result in incorrect measurements
- 6.2 Glassware not properly cleaned may result in fictitious results due to residue from previous experiments.
- 6.3 Platinum-iridium ring should be rinsed with benzene, followed by acetone and then flashed over a Bunsen burner to remove hydrocarbon residue.

7.0 Procedure

7.1 General Maintenance

- 7.1.1 Place tensiometer on a sturdy surface in an area that is free from of excessive vibration, air currents, and relative humidity.
- 7.1.2 Annual application of oil to the zero adjustment shaft and bearing should be done using a light oil.
- 7.1.3 Replacement of the torsion wire may be necessary due to accidental breakage.

7.2 Calibration of Tensiometer

- 7.2.1 Arrest the torsion arm by using the clamping mechanism located directly behind the index assembly.
- 7.2.2 Hang the platinum-iridium ring on the hook at the end of the lever.
- 7.2.3 Cut a small strip of paper and fit it onto the ring to act as a platform for a weight which will be used for calibration.
- 7.2.4 Release the torsion arm, and adjust the knob on the right side case until the index and its image are exactly in line with the reference line on the mirror.

- 7.2.5 Turn the knob beneath the main dial on the front of the instrument until the vernier reads zero on the outer scale of the dial.
- 7.2.6 Arrest the torsion arm as done in 6.2.1.
- 7.2.7 Place a known mass of 600mg for calibration on the paper platform.
- 7.2.8 Release the torsion arm.
- 7.2.9 Turn the knob on the right side of the case in a counterclockwise direction until the index and its image are again exactly in line with the reference line of the mirror.
- 7.2.10 Record the dial reading to the nearest 1/10 scale division.
- 7.2.11 Determine the accuracy of the calibration from the reading obtained. The apparent surface tension, S , is given as follows: $S = Mg/2L$
 - Where:
 - M = the weight expressed in grams = 0.600g
 - g = acceleration of gravity expressed in $\text{cm/sec}^2 = 980$
 - L = mean circumference of the ring in centimeters = ~ 6.0 cm
 - S = dial reading = 49.00 dynes/cm (expected value)
- 7.2.12 If the dial reading differs from the expected value, then the effective length of the torsion arm must be adjusted until these two values agree.
- 7.2.13 This adjustment is accomplished by turning the knurled knob at the left end of the lever arm. If the recorded dial reading is greater than the calculated value, move the hook to shorten the effective length of the arm. Conversely, if the dial reading is less than the calculated value, move the hook to lengthen the effective length of the arm.
- 7.3 Measuring Surface Tension From a Sample Liquid
 - 7.3.1 Arrest the torsion arm holding the cleaned platinum-iridium ring by locking the clamping mechanism.
 - 7.3.2 Place the sample liquid in a properly cleaned glass container and then onto sample table.
 - 7.3.3 Move the sample table around until it is directly beneath the platinum-iridium ring.

- 7.3.4 Raise the sample table until the ring is immersed in the sample liquid.
- 7.3.5 Release the torsion arm and adjust the knob on the right side of the case until the index and its image are exactly in line with the reference mark on the mirror.
- 7.3.6 Turn the knob beneath the main dial on the front of the case until the vernier reads zero on the outer scale of the dial.
- 7.3.7 Lower the sample table until the ring is in the surface of the liquid, adjusting the knob on the right side of the case to keep the index lined up with the reference mark on the mirror.
- 7.3.8 Continue the two simultaneous adjustments until the distended film at the surface of the liquid breaks.
- 7.3.9 The scale reading at the breaking point of the distended film is the apparent surface tension.
- 7.3.10 Using the Correction Factor Chart, located in the Instruction Manual, page number 9, determine the true surface tension.
- 7.4 Measuring Interfacial Tension From a Dense Liquid to a Lighter Liquid
 - 7.4.1 Arrest the torsion arm holding the cleaned platinum-iridium ring by locking the clamping mechanism.
 - 7.4.2 Place the more dense sample liquid in a properly cleaned glass container and then onto the sample table.
 - 7.4.3 Move the sample table around until it is directly beneath the platinum iridium ring.
 - 7.4.4 Raise the sample table until the ring is immersed in the sample liquid.
 - 7.4.5 Release the torsion arm and adjust the knob on the right side of case until the index and its image are exactly in line with the reference mark on the mirror.
 - 7.4.6 Turn the knob beneath the main dial on the front of the case until the vernier reads zero on the outer scale of the dial.
 - 7.4.7 Carefully pour the lighter liquid onto the surface of the heavier liquid to a

depth of about $\frac{1}{4}$ to $\frac{1}{2}$ inch.

- 7.4.8 Allow the interface to age for a period of about 30 seconds, but remember to log this during the experiment.
- 7.4.9 Lower the sample table until the ring is in the interface between the two liquids, adjusting the knob on the right side of the case to keep the index lined up with the reference mark on the mirror.
- 7.4.10 Continue the two simultaneous adjustments until the distended film at the interface ruptures.
- 7.4.11 The scale reading at the breaking point of the interfacial film is the apparent interfacial tension.
- 7.4.12 Using the Correction Factor Chart, located in the Instruction Manual, page number 9, determine the true interfacial tension.
- 7.5 Measuring Interfacial Tension From a Lighter Liquid to a More Dense Liquid
 - 7.5.1 Place the more dense sample liquid in a properly cleaned glass container. The layer should be deep enough so that the ring will not contact the bottom of the container before the interface ruptures.
 - 7.5.2 Carefully pour the lighter liquid onto the surface of the heavier liquid already in the container.
 - 7.5.3 Arrest the torsion arm holding the cleaned platinum-iridium ring by locking the clamping mechanism.
 - 7.5.4 Move the sample table around until it is directly beneath the platinum-iridium ring.
 - 7.5.5 The sample table is now raised until the ring is immersed in the lighter liquid.
 - 7.5.6 Release the torsion arm and adjust the instrument to a zero reading.
 - 7.5.7 Adjust the knob on the right side of the case until the index and its image are exactly in line with the reference mark on the mirror.
 - 7.5.8 Turn the knob beneath the main dial on the front of the case until the vernier reads zero on the inner scale of the dial.

- 7.5.9 Raise the sample table until the ring is in the interface between the two liquids, adjusting the knob on the right side of the case to keep the index lined up with the reference mark on the mirror.
- 7.5.10 Continue the two simultaneous adjustments until the distended film at the interface ruptures.
- 7.5.11 The scale reading at the breaking point of the interfacial film is the apparent interfacial tension.
- 7.5.12 Using the Correction Factor Chart, located in the Instruction Manual, page number 9, determine the true interfacial tension.

8.0 QA/QC

- 8.1 The tensiometer should be uniquely identified.
- 8.2 Tensiometers shall be calibrated before use if there is an extended time period between experiments such as a month or on a weekly basis if being used regularly.
- 8.3 A lab notebook should be kept and updated concerning the maintenance and calibration of the tensiometer.
- 8.4 Samples are measured three times and the average is taken.
- 8.5 Water should be measured in order to distinguish if tensiometer is measuring accurately.

Water	10C	20C	30C	40C	50C	60C	70C	80C	90C
Viscosity*	74.8	73.6	71.8	70.1	68.2	66.8	65.0	63.0	61.2

* viscosity is measured in dynes per centimeter

It should be noted that small solids in tap water and organics in RO water can affect the surface tension of water, for this reason measurements must agree with the expected value to +/-20%.

9.0 Calculations

Multiplication is used to find the actual tension measurements by taking the correction factor, located on page 9 of the instruction manual, and multiplying it by the apparent tension measurement first obtained. The surface tension will be in units of dynes/cm.

10.0 Miscellaneous Notes

N/A

11.0 References

Fisher Surface Tensiometer Model 20 Instruction Manual

APPENDIX B3

Standard Operating Procedure

Brookfield Dial Reading Viscometer

Disclaimer:

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1.0 Purpose (Scope and Application)

To discover the torque necessary to overcome the viscous resistance exerted on a sensing element using the Brookfield Dial Reading Viscometer with either the small sample adapter or the Ultra low (UL) adapter. The range of viscosity measurements depends upon sample adapter and spindle type. The measurements are made at a constant temperature ranging from 5 degrees centigrade to 90 degrees centigrade.

2.0 Method Summary

A liquid sample is placed in a container. This container is placed inside a water jacket, when a constant temperature is necessary. The viscometer is then turned on and ran with a constant speed. After the needle has stabilized on the dial the measurement is recorded by depressing the clutch and turning off the motor. The reading is corrected using a correction chart, which is spindle specific, provided by the instruction manual.

3.0 Reagents

N/A

4.0 Equipment and Apparatus

Brookfield Dial Reading Viscometer with Small Sample Adapter or UL Adapter

5.0 Health and Safety Precautions

5.1 When using the water bath, caution needs to be taken to prevent burns from high temperatures.

5.2 When testing volatile liquids, the viscometer and water bath need to be setup inside the hood to prevent exposure.

6.0 Interferences

6.1 Once a sample has been placed inside the water bath, 1 hour should be allowed for equilibration. If the sample is not equilibrated to temperature the viscosity will be inaccurate.

6.2 Dial readings under 10 have a reduced accuracy, where as accuracy improves as the reading approaches 100 with the Brookfield Dial Reading Viscometer.

6.3 When pouring a sample liquid into the sample adapter be sure no air is trapped under the liquid for this may cause a discrepancy in the reading.

6.4 The viscometer must be leveled using the bubble level located on the stand to ensure accuracy.

7.0 Procedure

7.1 General Maintenance

7.1.1 Care must be taken to prevent dust, fumes, liquids, and other forms of contamination from entering the viscometer housing.

7.1.2 Never place the instrument upside down with a fluid-coated spindle attached.

7.1.3 Do not expose the viscometer to ambient temperatures in excess of 75 Degrees C.

7.1.4 Avoid applying side or down thrust to the spindle coupling.

7.2 Calibration of Viscometer

7.2.1 Mount and level the viscometer with no spindle attached. The power switch should be off.

7.2.2 Turn the spindle coupling to deflect the pointer from its zero position to a reading of 5 to 10 and let it swing back under its own power.

- 7.2.3 If the pointer swings freely and smoothly, and returns to zero each time this is repeated, the pivot point and jewel bearing are in good condition.
- 7.2.4 The use of a calibrated viscosity standard should be used as a final check for performance. Cole-Parmer viscosity standards N1.0 and S20 are used calibration checks.

For the Ultra Low Adapter, use N1.0 viscosity standard.

Viscosity standard	20 C	25 C	37.78 C	40C	50C
N1.0 (mPa's, cP)	1.001	0.9277	0.7753	0.7531	0.6642

For the Small Sample Adapter, use S20 viscosity standard.

Viscosity Standard	20 C	25 C	38.8 C	40 C	50 C	98.9 C	100 C
S20 (mPa's, cP)	35.06	27.69	16.25	14.95	10.59	3.187	3.123

- 7.2.5 Measurement of the viscosity standard should be accurate to +/-10% of the expected value. If calibration check fails, make sure fluid is equilibrated, the spindle is immersed to the immersion mark, the viscometer is level, and without spindle attached a reading of zero is obtained. If calibration check fails after checking these items the viscometer must be serviced.
- 7.3 Setup of the Brookfield Viscometer with Small Sample Adapter and the No.18 Spindle for Temperature Dependant Measurement, for Liquids with Viscosities in the Range of 5-10,000 centipoise (cp)
- 7.3.1 Mount the viscometer securely on the stand.
- 7.3.2 Attach locating channel to viscometer by threading the upper mounting screw into the tapped hole in the viscometer pivot cup.
- 7.3.3 Attach the water jacket assembly to the locating channel with the lower mounting screw.
- 7.3.4 Connect hose from temperature bath outlet to the lower fitting on the water jacket. The water bath should be set at desired temperature.
- 7.3.5 Connect hose from temperature bath inlet to the upper fitting on the water
- 7.3.6 Load the removable sample chamber with 8 ml of sample fluid.
- 7.3.7 Carefully guide sample chamber into water jacket from the bottom until it contacts the metal pin in the jacket top plate. Rotate chamber until the pin engages the slot in the side of the chamber. Raise chamber and rotate

counter-clockwise until it stops. Release chamber, allowing it to drop and lock into place.

- 7.3.8 Assemble the spindle, extension link, and coupling nut. Slowly lower Spindle into sample liquid and thread coupling nut onto viscometer.
- 7.3.9 Level the viscometer using the bubble level on viscometer stand.
- 7.4 Setup of the Brookfield Viscometer with Ultra Low (UL) Adapter for Temperature Dependant Measurement, For Liquids with Viscosities in the Range of 1-2000cp
 - 7.4.1 Mount the viscometer securely on the stand.
 - 7.4.2 Attach locating channel to viscometer by threading the upper mounting screw into the tapped hole in the viscometer pivot cup.
 - 7.4.3 Assemble the spindle, extension link, and coupling nut. Thread coupling nut onto viscometer.
 - 7.4.4 Guide the UL tube over the spindle from underneath until locating pin seats in tube collar locating slot.
 - 7.4.5 Holding tube in place against locating channel, push lower mounting screw in, and thread into the tube collar.
 - 7.4.6 Immerse the tube in sample fluid up to the immersion groove by lowering the viscometer on the viscometer stand.
 - 7.4.7 Level the viscometer using the bubble level on the viscometer stand.
- 7.5 Making a Viscosity Measurement with Brookfield Viscometer
 - 7.5.1 With setup complete and sample properly positioned turn the motor switch on.
 - 7.5.2 Allow for the indicated reading to stabilize. This depends on speed and the properties of the sample fluid. Speed is selected to give dial reading within range.
 - 7.5.3 When making measurements at high speeds it will be necessary to depress the clutch and turn the motor off to view the pointer.
 - 7.5.4 After measurement is taken it must be multiplied by a correction factor that is specific to that spindle. The correction factors are provided by the

Assembly and Operating Instructions for both the Small Sample and UL
Adapters.

8.0 QA/QC

- 8.1 The viscometer should be uniquely identified.
- 8.2 Viscometer shall be checked for calibration before use if there is an extended time period between experiments such as a month or on a weekly basis if being used regularly.
- 8.3 A lab notebook should be kept and updated concerning the maintenance and calibration of the viscometer.
- 8.4 Samples are measured three times and the average is taken.
- 8.5 Measurement of the viscosity standard should be accurate to +/-10% of the expected value. If calibration check fails, make sure fluid is equilibrated, the spindle is immersed to the immersion mark, the viscometer is level, and without spindle attached a reading of zero is obtained. If calibration check fails after checking these items the viscometer must be serviced.

9.0 Calculations

Multiplication is used to find the viscosity by taking the dial reading and multiplying it by the correction factor. The viscosity is given in Centipoises.

10.0 Miscellaneous Notes

N/A

11.0 References

- 11.1 Brookfield Dial Reading Viscometer and Brookfield Digital Viscometer Model DV-I Operating Instructions. Manual No. M/85-150-D.
- 11.2 More Solutions to Sticky Problems: A Guide to Getting More From Your Brookfield Viscometer.
- 11.3 Small Sample Adapter Assembly & Operating Instructions
- 11.4 UL Adapter Assembly & Operating Instructions